Optimization of non-evaporable getter coating for accelerator beam pipe

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Outlet

- Introduction
- Pumping property
  - Film deposition
  - Surface analysis
  - NEG activation procedure
  - Pumping properties measurements
  - Activation temperature
  - Sticking probability and capacity for different NEG coatings
- Desorption properties
  - How to reduce ESD
  - What film is needed
  - What achieved
- Conclusions

What are usual considerations for vacuum

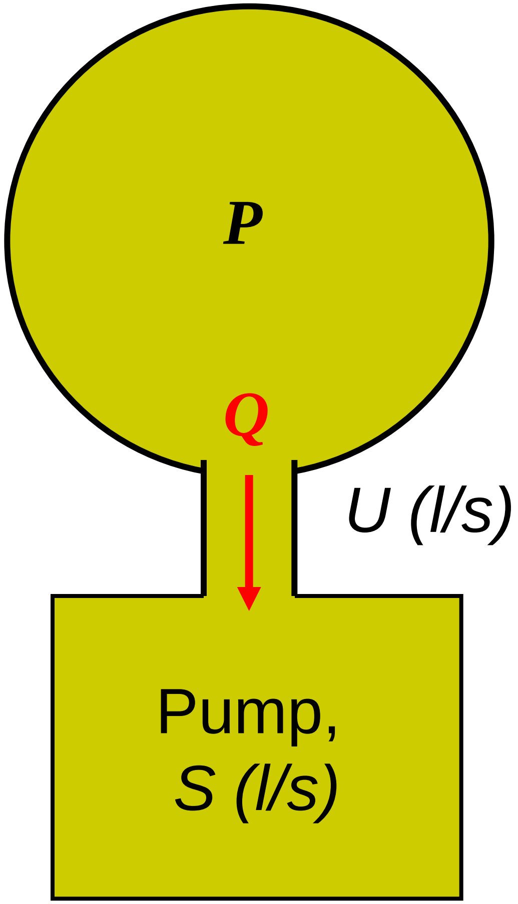
Required pressure P is defined by gas desorption Q in the vessel and effective pumping speed S_{eff}.

In a simple case it is

$$P = \frac{Q}{S_{eff}} = Q\left(\frac{1}{S} + \frac{1}{U}\right)$$

$$Q = qA + \eta_\gamma \Gamma + \eta_e \mathrm{I}_e + \eta_{ion} \mathrm{I}_{ion}$$

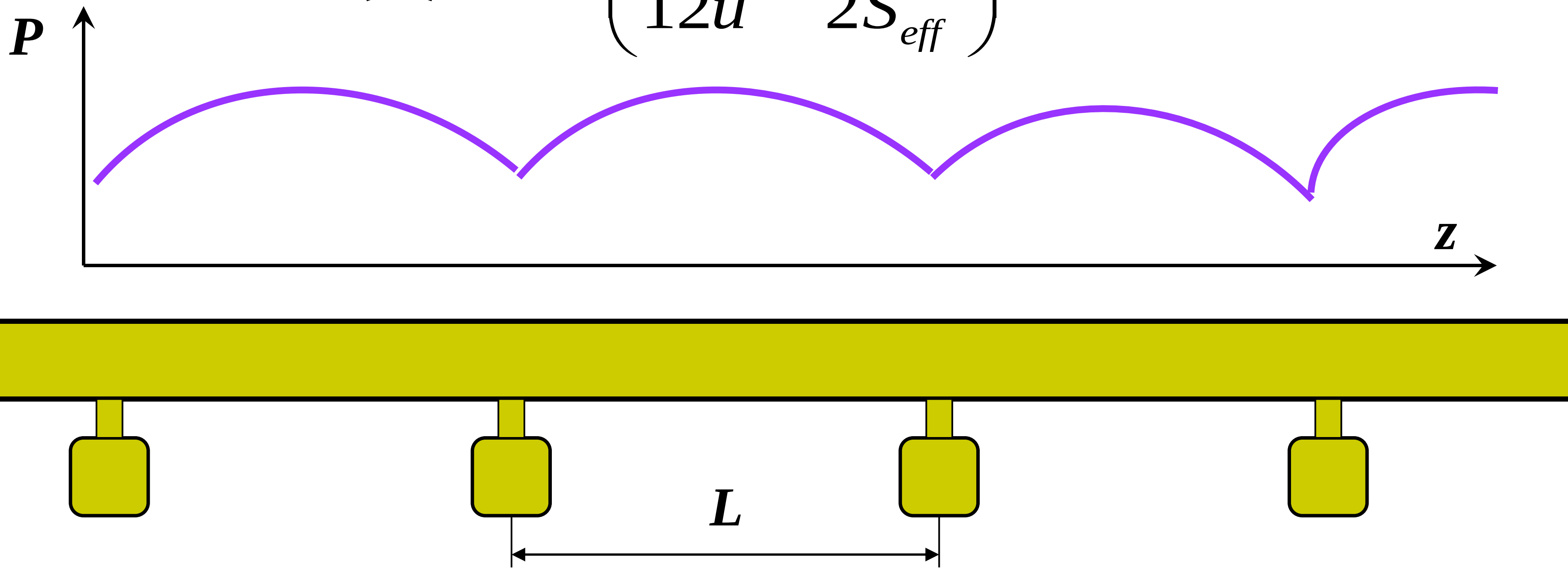
Thermal, photon, electron and ion stimulated desorption



Usual accelerator vacuum chamber

- Long tube with length ***L* >> *a*,** where *a* - transversal dimension
- Average pressure depends on vacuum conductance ***u(L,a)*** of the beam vacuum chamber

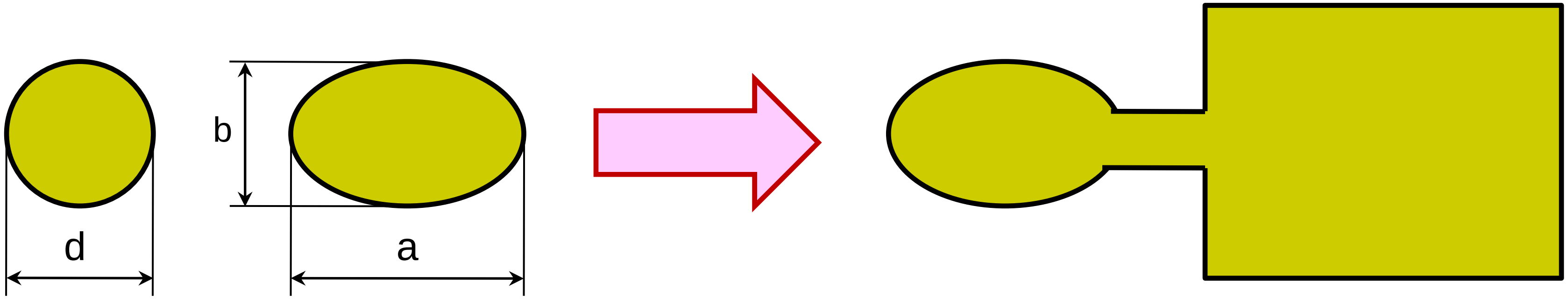
$$\langle P \rangle = qL\left(\frac{L}{12u} + \frac{1}{2S_{eff}}\right)k_B T$$



Vacuum chamber cross sections

Beam pipe
Circular or elliptical
4 mm ≤ d, a, b ≤ 200 mm

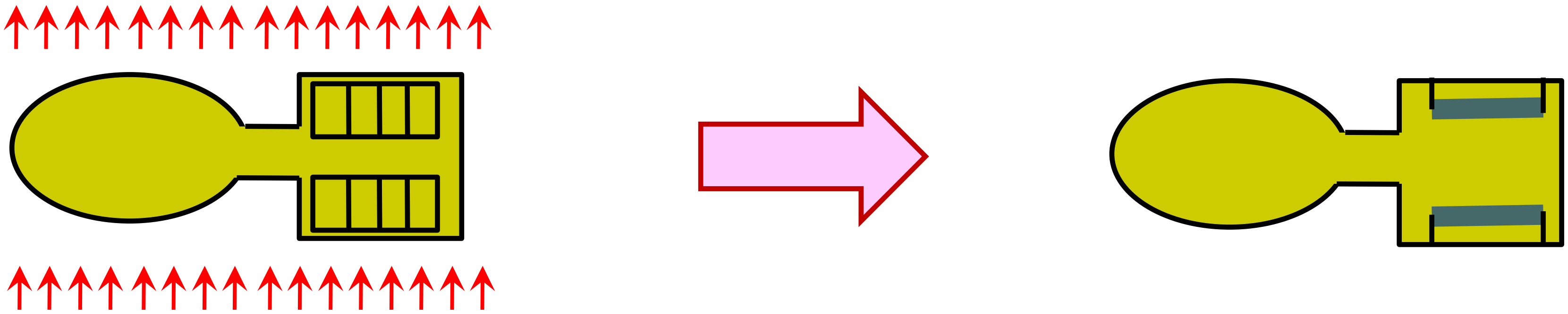
Vacuum chamber with an antechamber
for larger vacuum conductance, *U*



Distributed pumping

In dipole magnetic field

With NEG strips
(LEP in CERN)



Two concepts of the ideal vacuum chamber

Traditional:

- surface which outgasses as little as possible (‘nil’ ideally)
- surface which ***does not pump*** otherwise that surface is contaminated over time

Results in

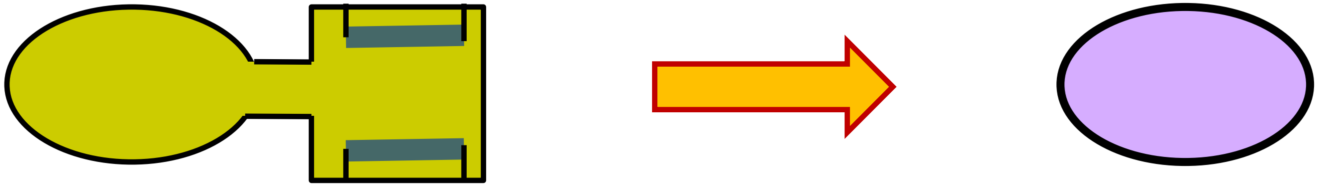
- Surface cleaning, conditioning, coatings
- Vacuum firing, *ex-situ* baling
- Baking *in-situ* to up to **300°C**
- Separate pumps

‘New’ (C. Benvenuti, CERN, ~1998):

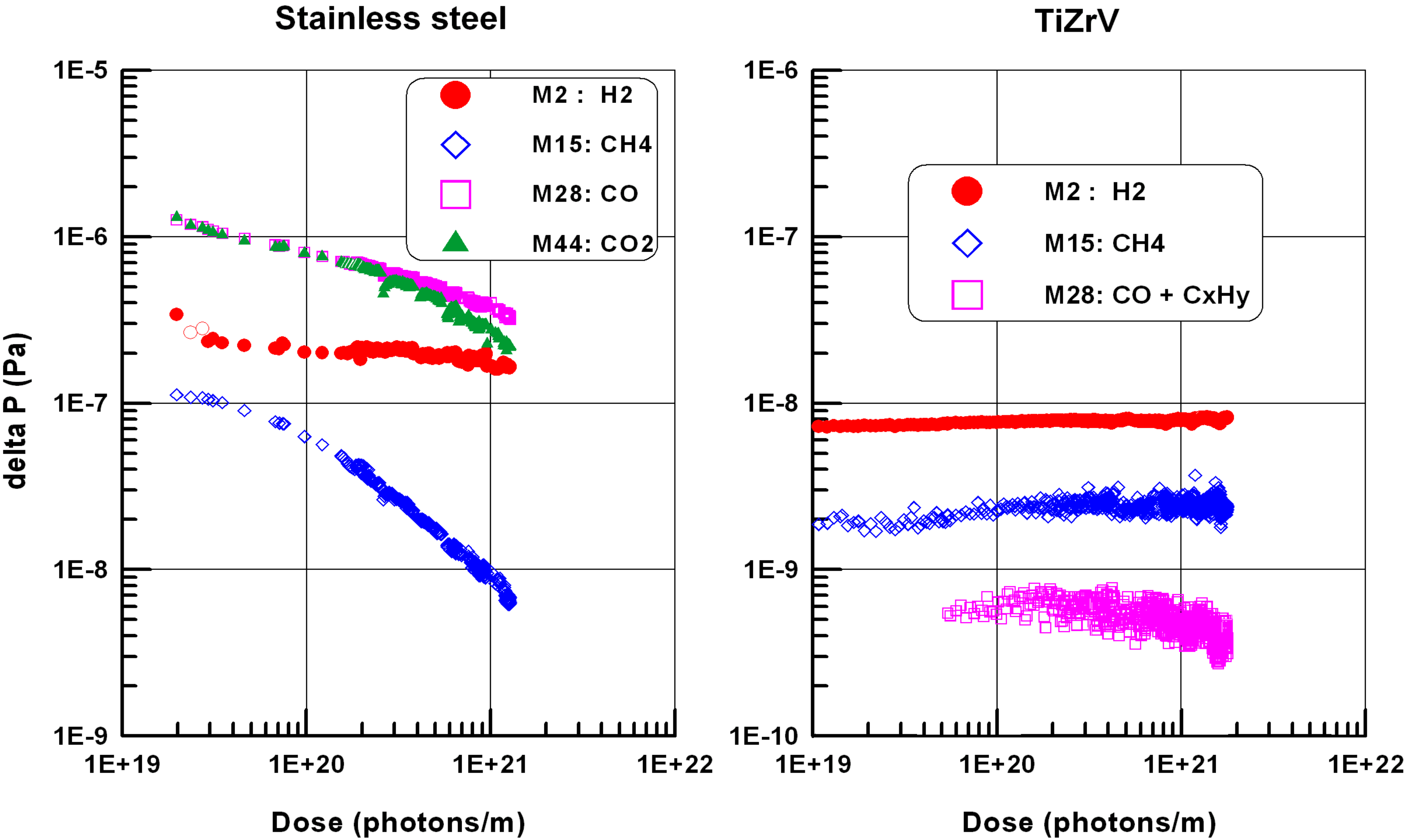
- surface which outgasses as little as possible (‘nil’ ideally)
- a surface which ***does pump***, however, will not be contaminated due to a very low outgassing rate

Results in

- NEG coated surface
- There should be no un-coated parts
- Activating (baking) *in-situ* at **150-180°C**
- Small pumps for C_xH_y and noble gases



Stainless steel vs. NEG coated vacuum chamber under SR



V.V. Anashin et al. Vacuum 75 (2004), p. 155.

NEG coating for accelerators

- First used in the ESRF (France);
- ELETTRA (Italy);
- Diamond LS (UK);
- Soleil (France) – first fully NEG coated;
- LHC (Switzerland) – longest NEG coated vacuum chamber;
- SIS-18 (Germany);
- and many others.
- NEG film capacity for CO and CO_2 is ~1ML:
  - If $P = 10^{-9}$ mbar then 1 ML can be sorbed just in ~10^3 -10^4 s;
  - Lab measurements of different NEG coatings often don't repeat CERN's data on sticking probability and capacity;
  - However, NEG coated parts of accelerators work well.

NEG coating for accelerators (2)

- What is required:
  - Input data for accelerator design:
    - $\eta(D,E,T_a)$, $\alpha(M, T_a)$, pumping capacity;
  - Better understanding:
    - what and why;
    - practical ‘do’s and ‘don’t’s;
  - Further development of this coating:
    - lower η, T_a, SEY;
    - higher $\alpha(M)$, pumping capacity;
    - optimising for an application.

What NEG coating does

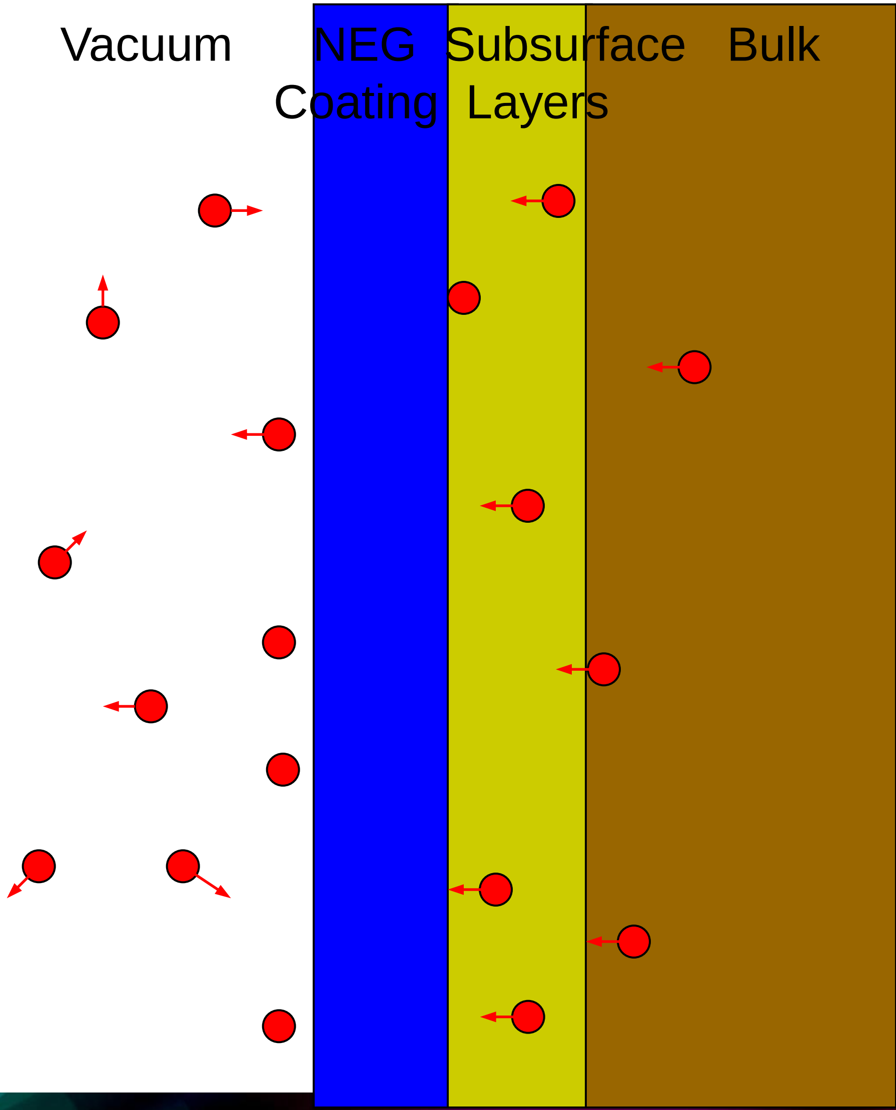
- **Reduces gas desorption:**
  - **A pure metal film ~1-μm thick without contaminants.**
  - **A barrier for molecules from the bulk of vacuum chamber.**
- **Increases distributed pumping speed, *S*:**
  - **A sorbing surface on whole vacuum chamber surface**

$S = \alpha \cdot A \cdot v/4$;

where **α – sticking probability,**

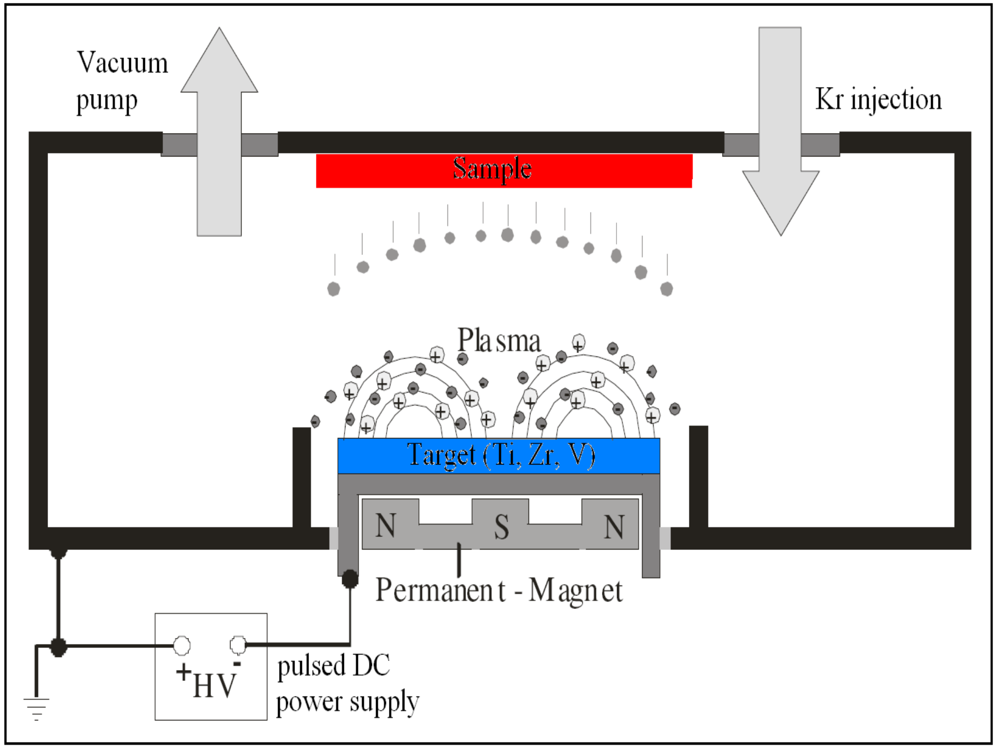
A – surface area,

v – mean molecular velocity

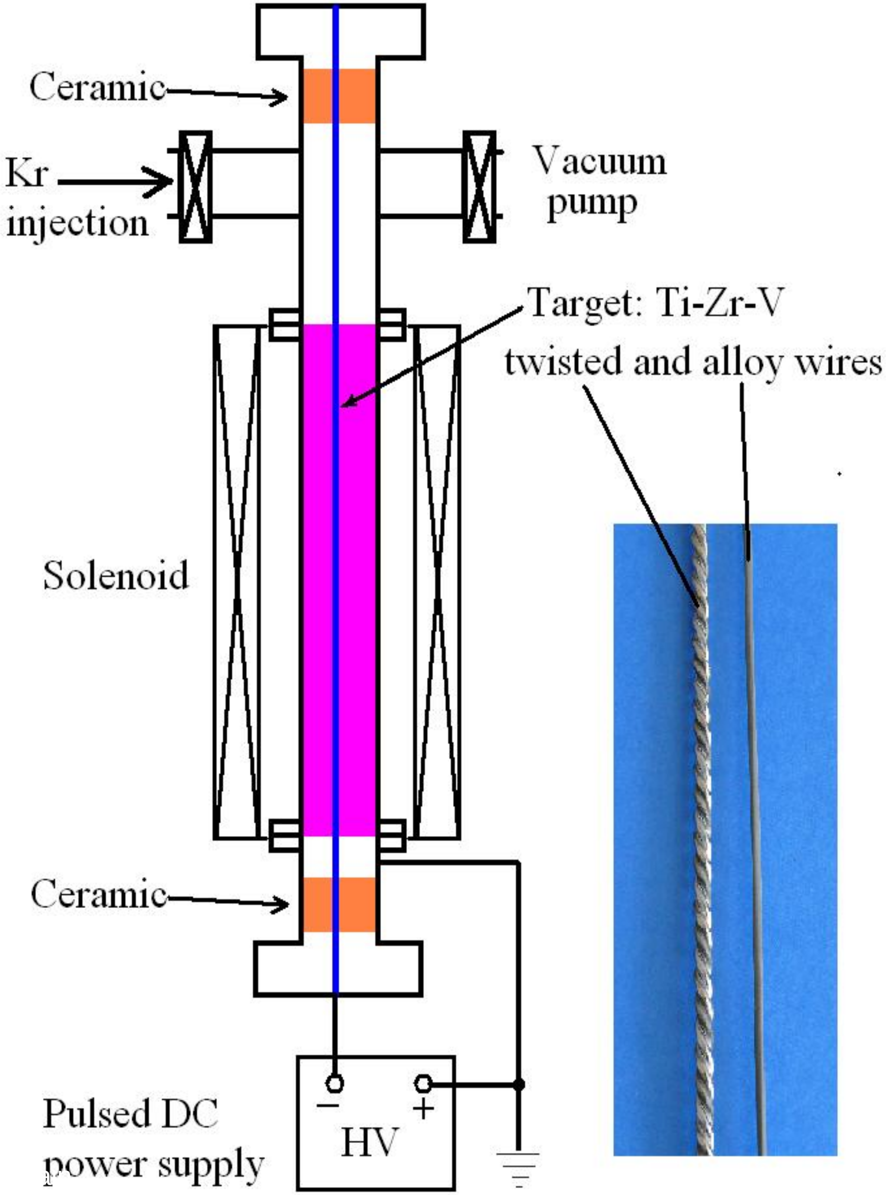


Deposition method

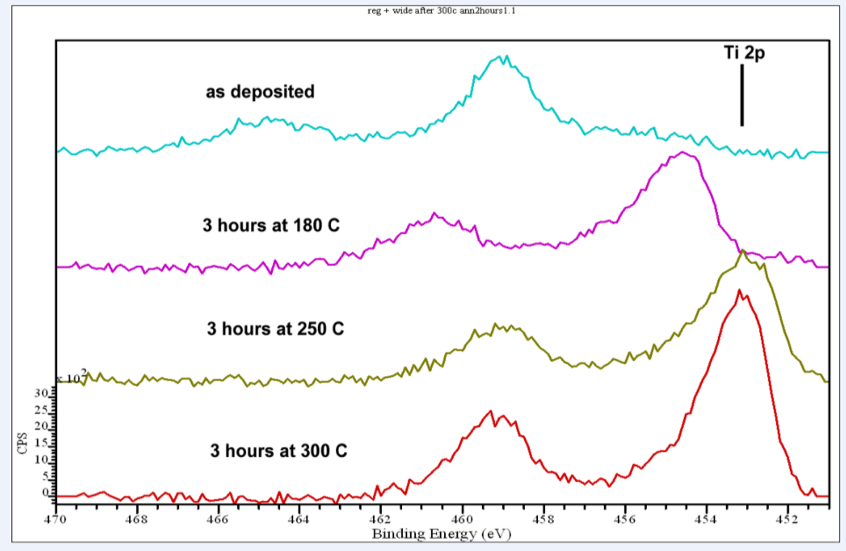
Planar magnetron deposition

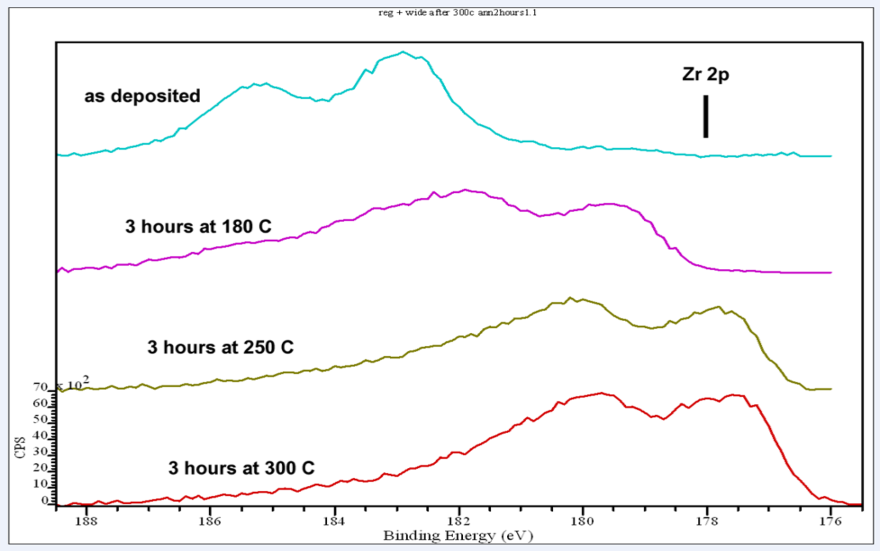


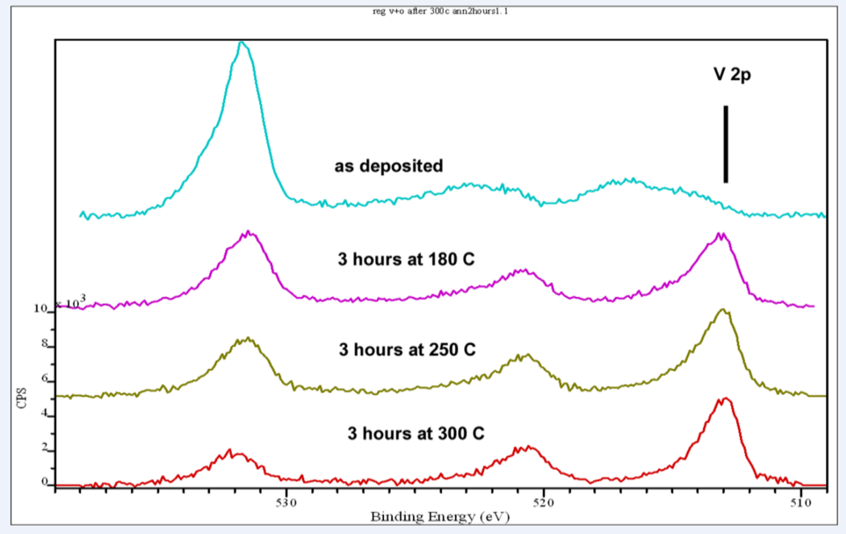
Cylindrical magnetron deposition

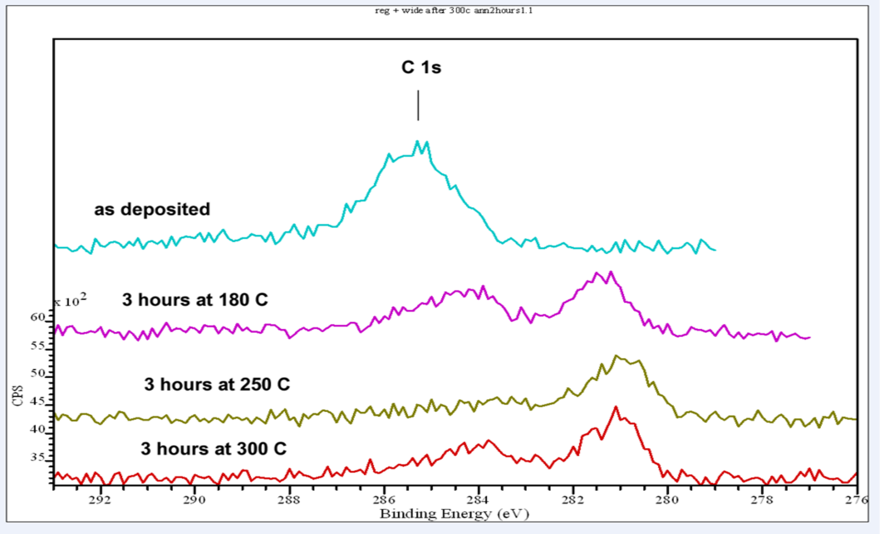


Region scan of XPS core levels of Ti, Zr, C and V of a Ti-Zr-V film (surface composition and chemical bounding)

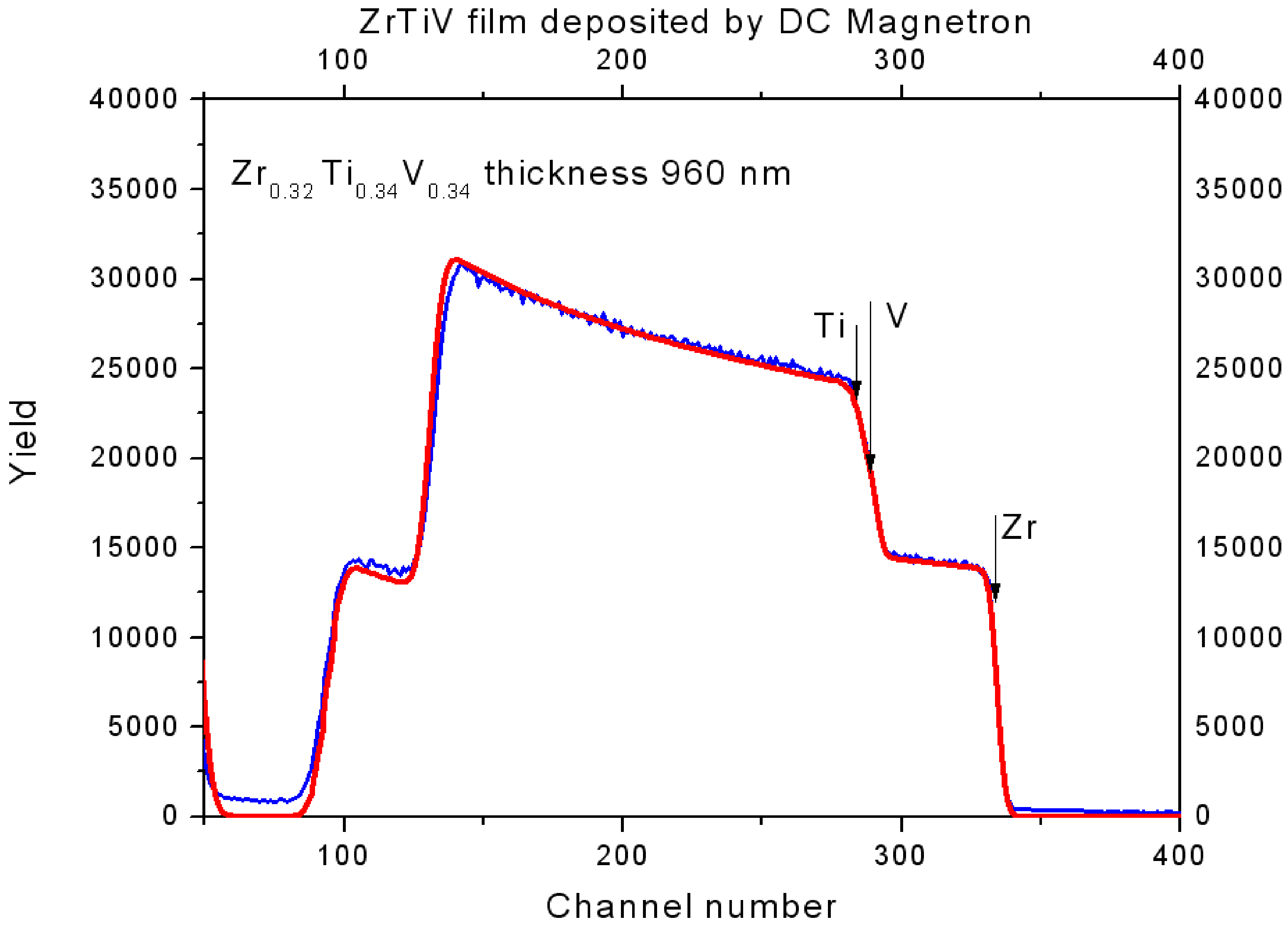




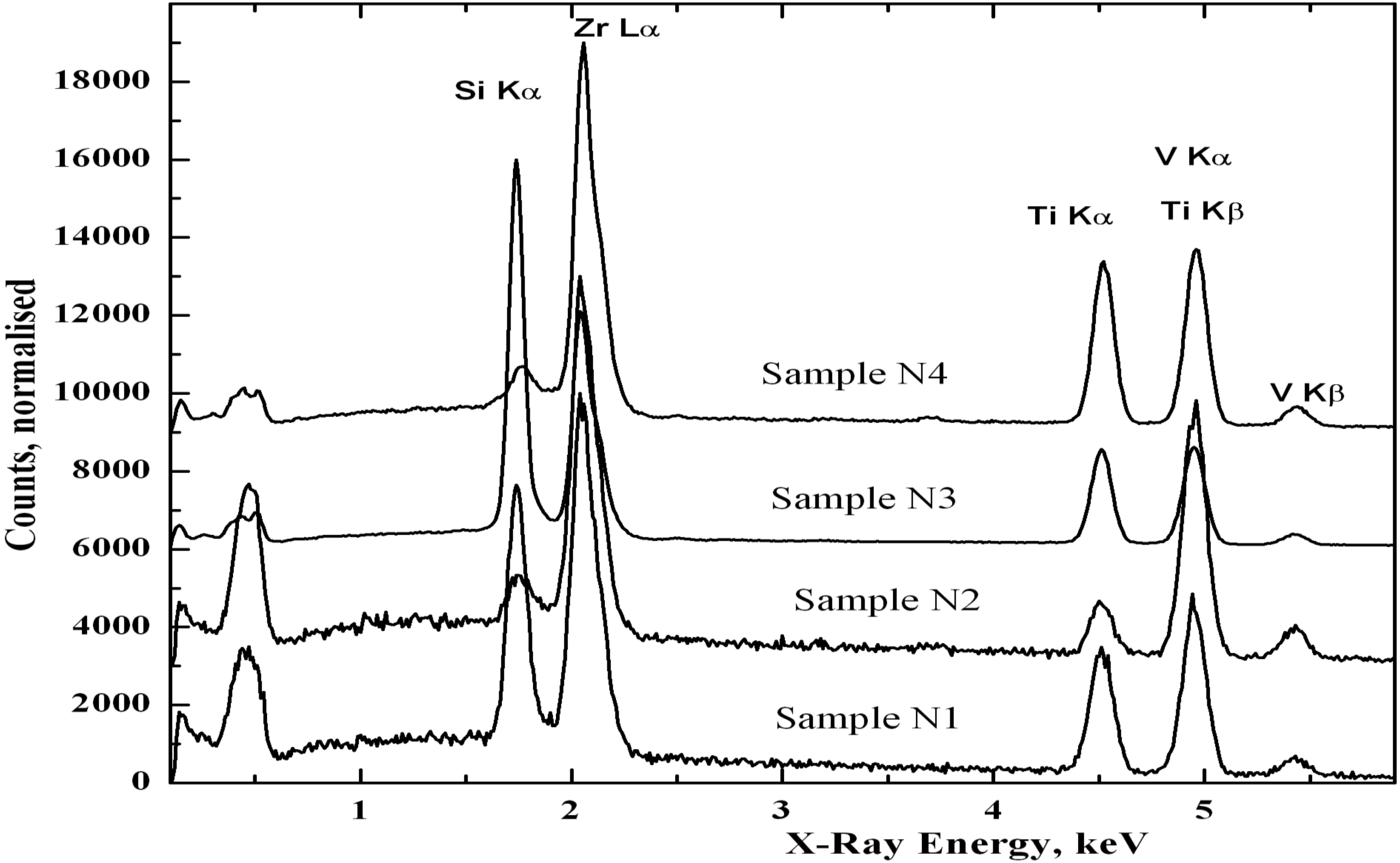




RBS (film compositions in bulk)

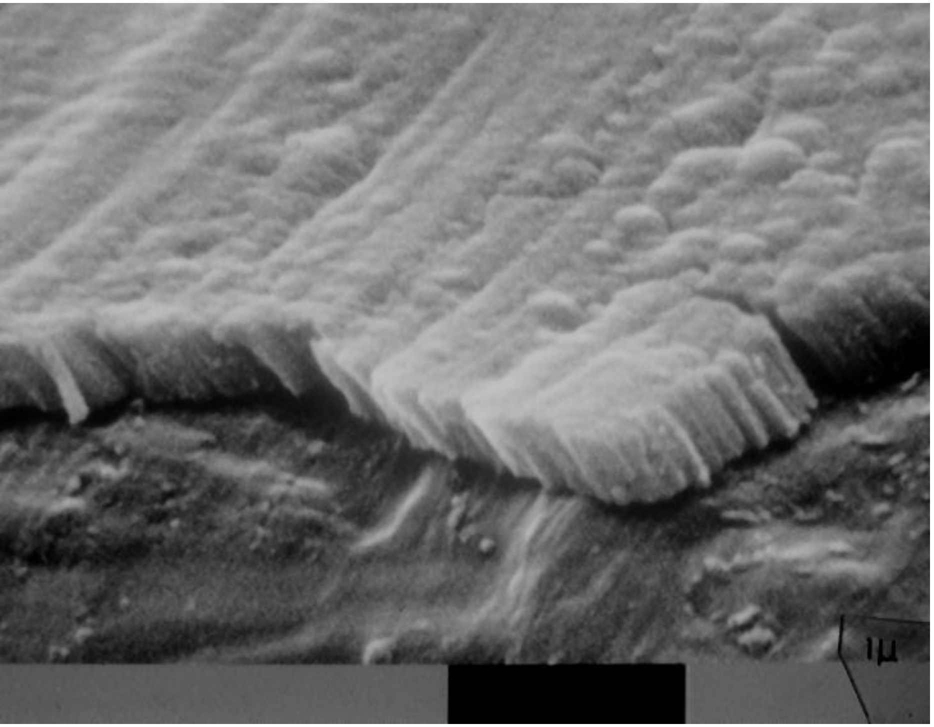


The EDX analysis for determination of film composition

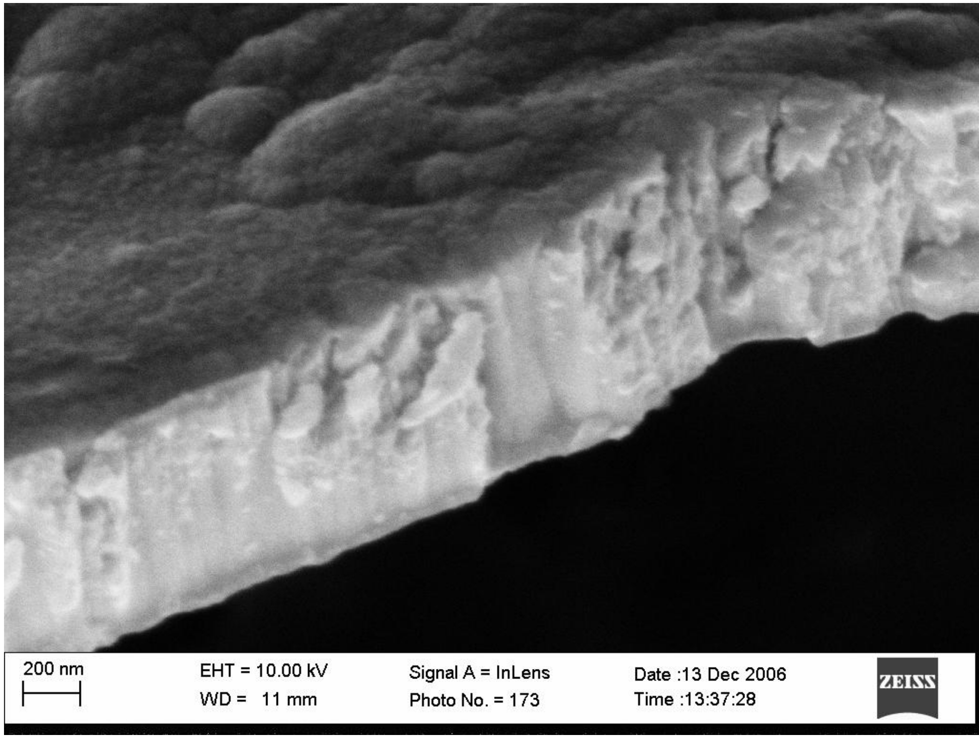


SEM images of films (film morphology)

columnar

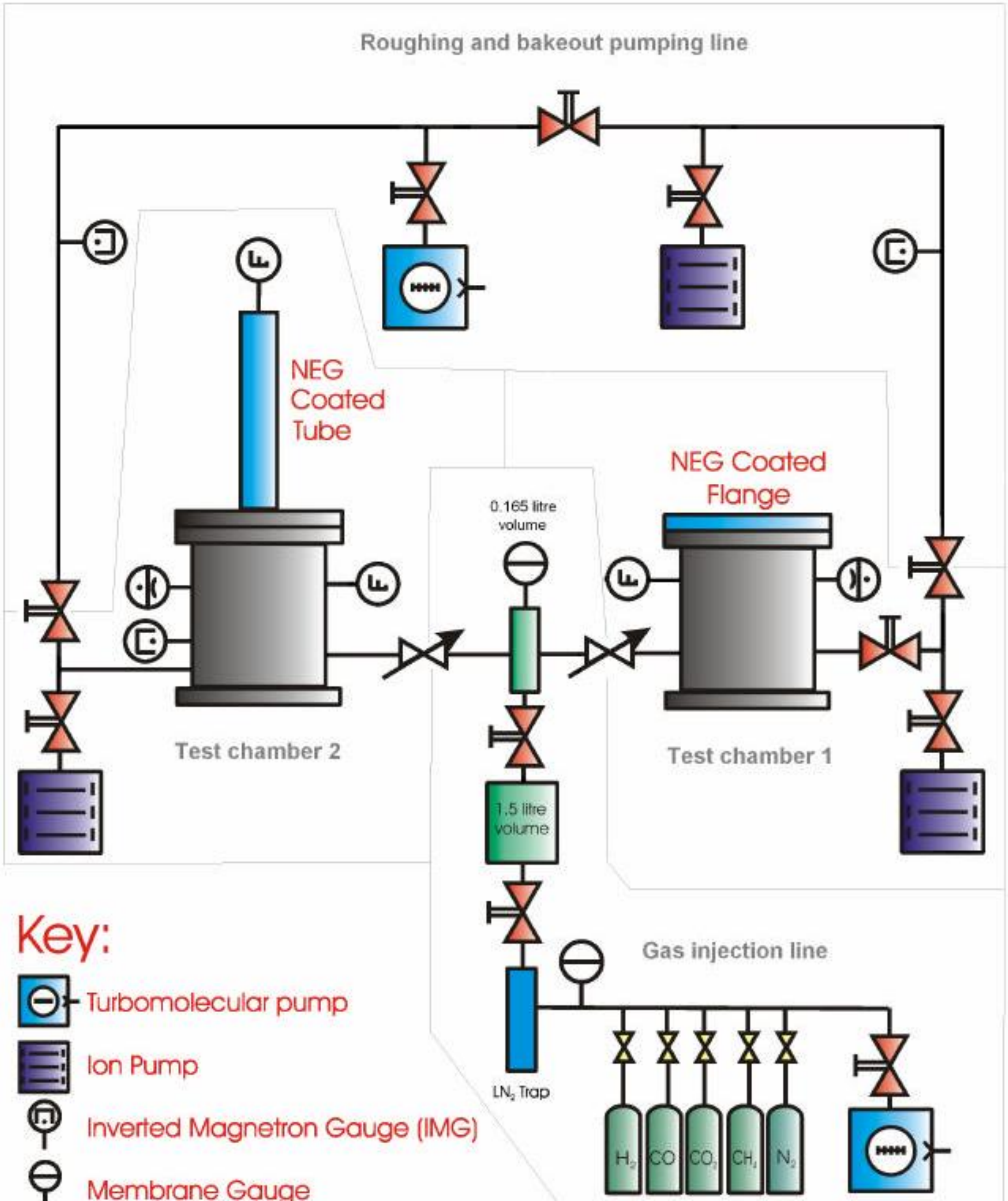


dense



O.B. Malyshev, R. Valizadeh, J.S. Colligon *et al*. J. Vac. Sci. Technol. A 27 (2009), p. 521.

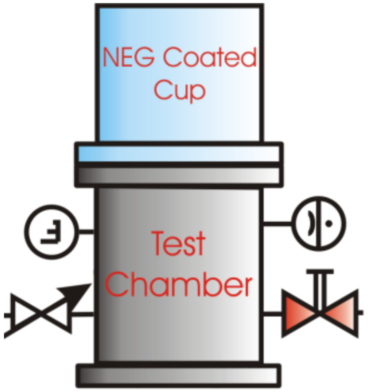
Set-up for NEG pumping evaluation



Key:
- Turbomolecular pump
- Ion Pump
- Inverted Magnetron Gauge (IMG)
- Membrane Gauge

O.B. Malyshev and K.J. Middleman.
Vacuum 83 (2009), p. 976.

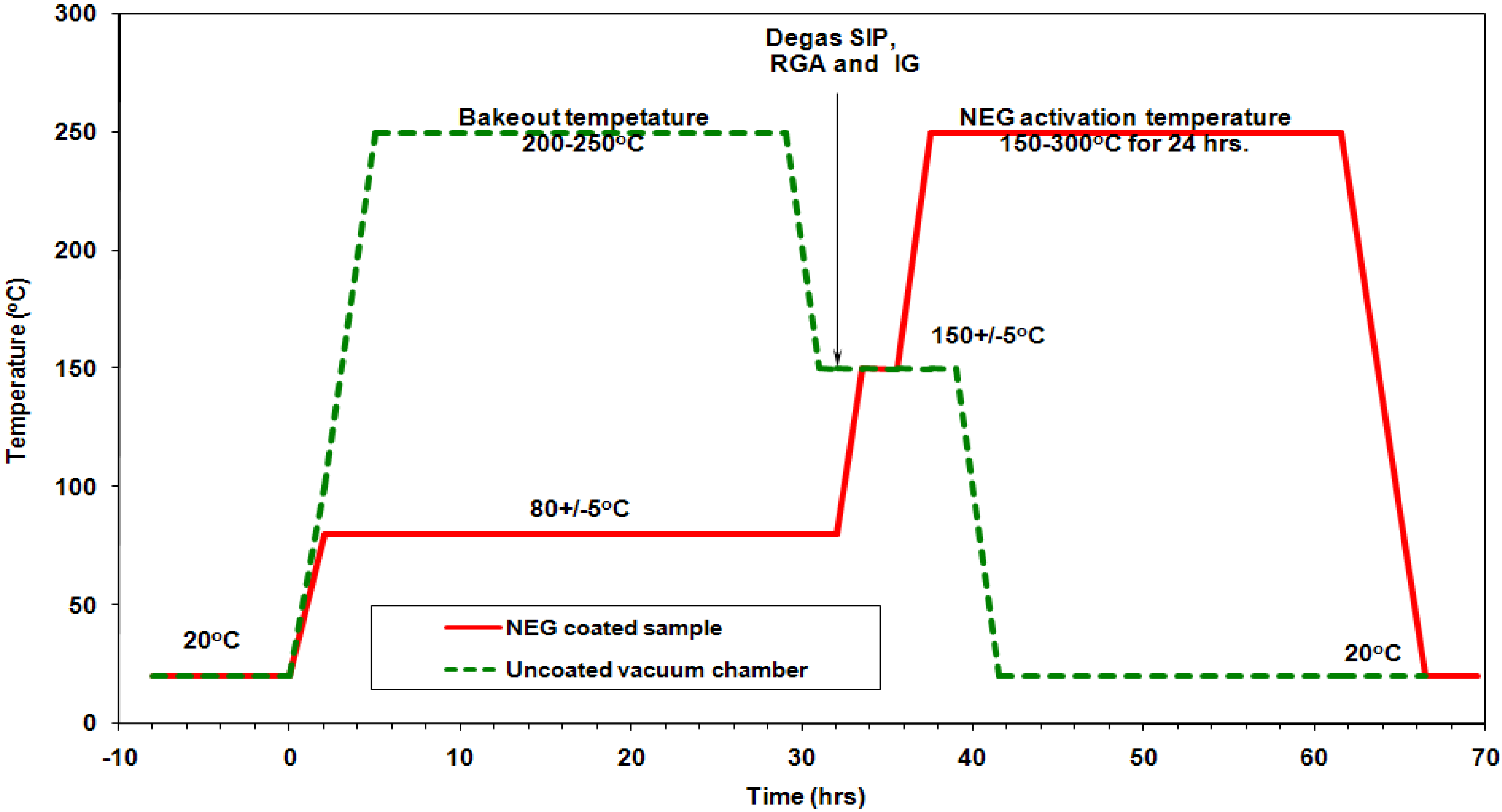
O.B. Malyshev et al.
J. Vac. Sci. Technol. A 27 (2009), p. 321.



Test chamber 1
(option)

- Residual Gas Analyser (RGA)
- Extractor Gauge
- Fine Leak Valve

ASTeC activation procedure



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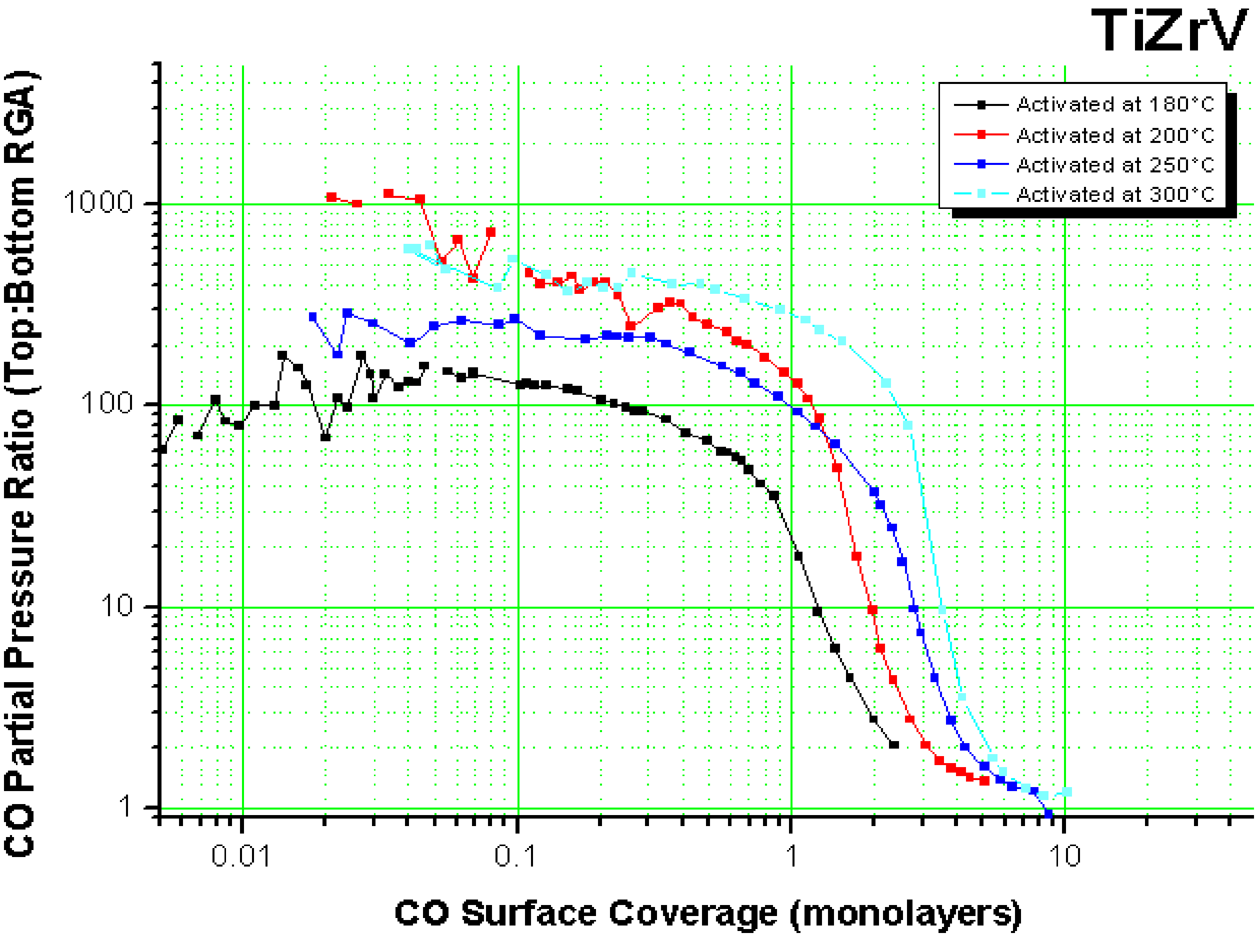
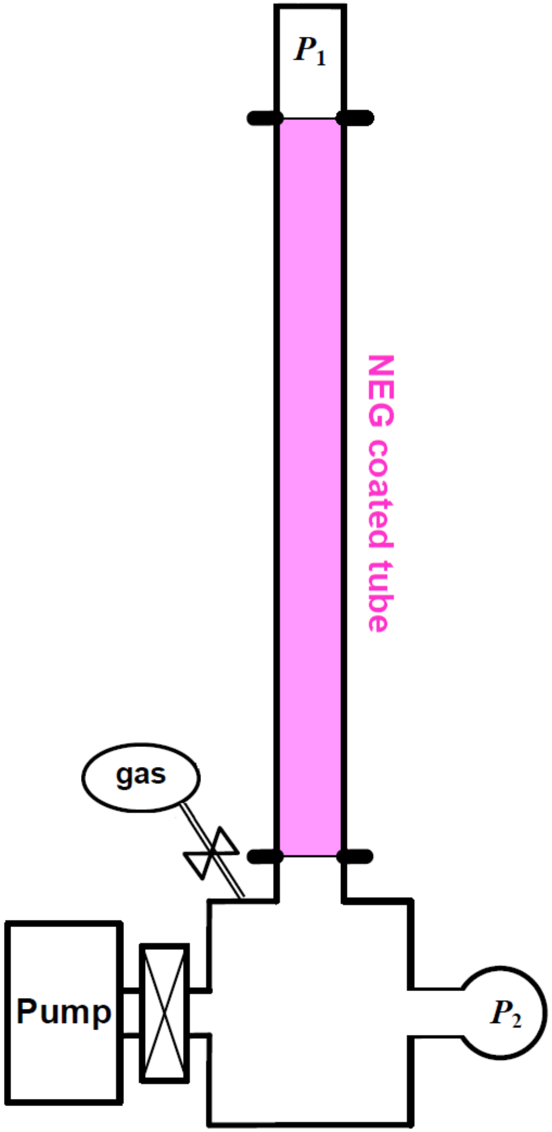
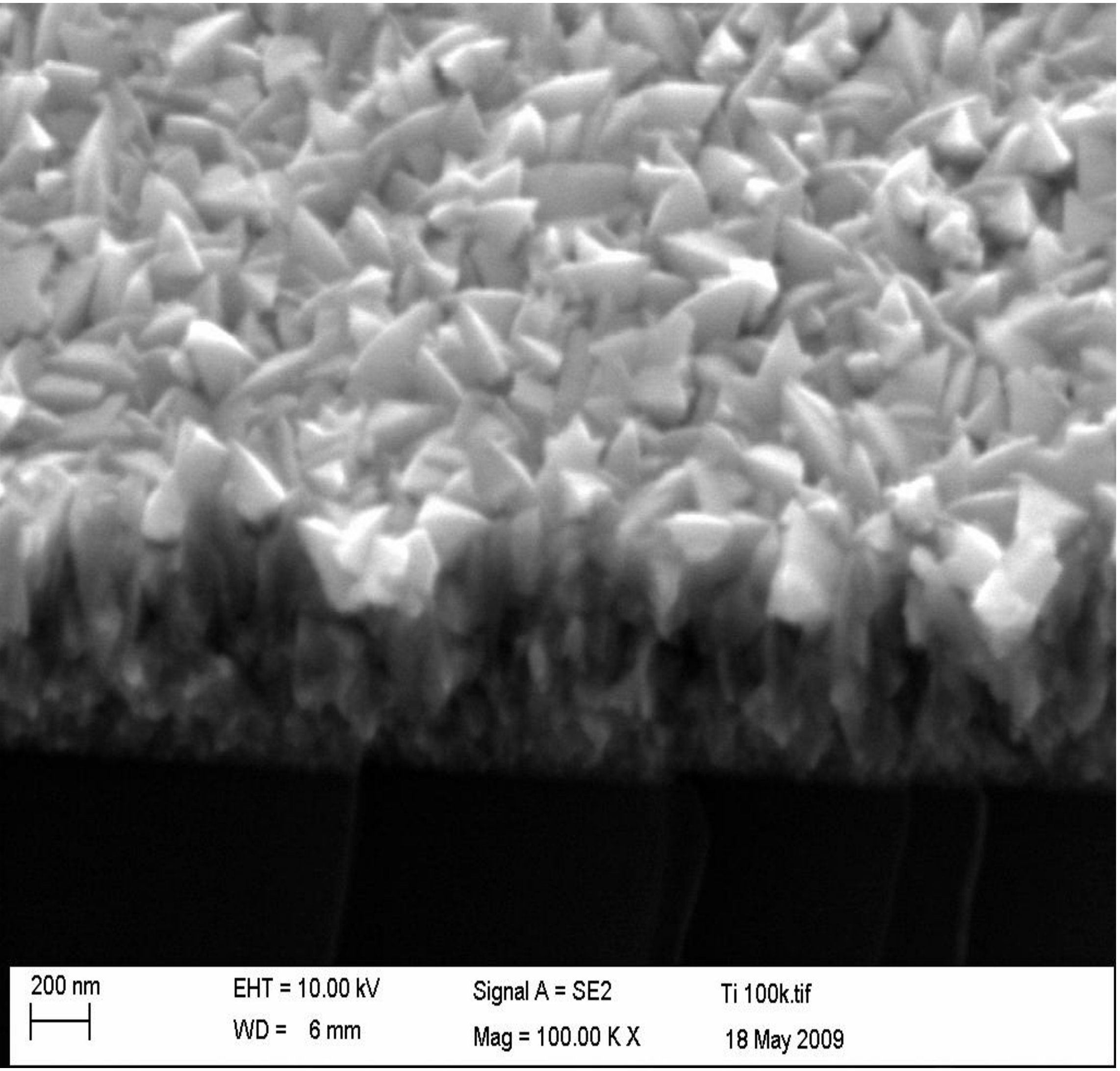
NEG pumping properties





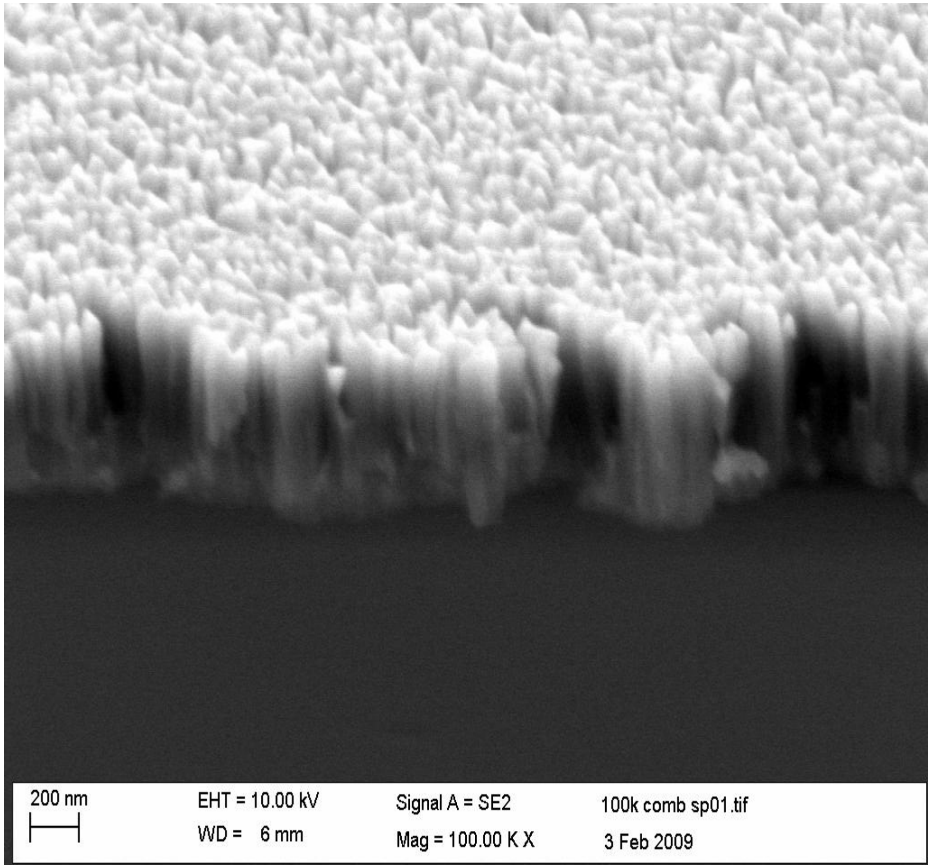
Pressure ratio P1/P2 measured during gas injection is used to estimate: initial sticking probability and sorption capacity

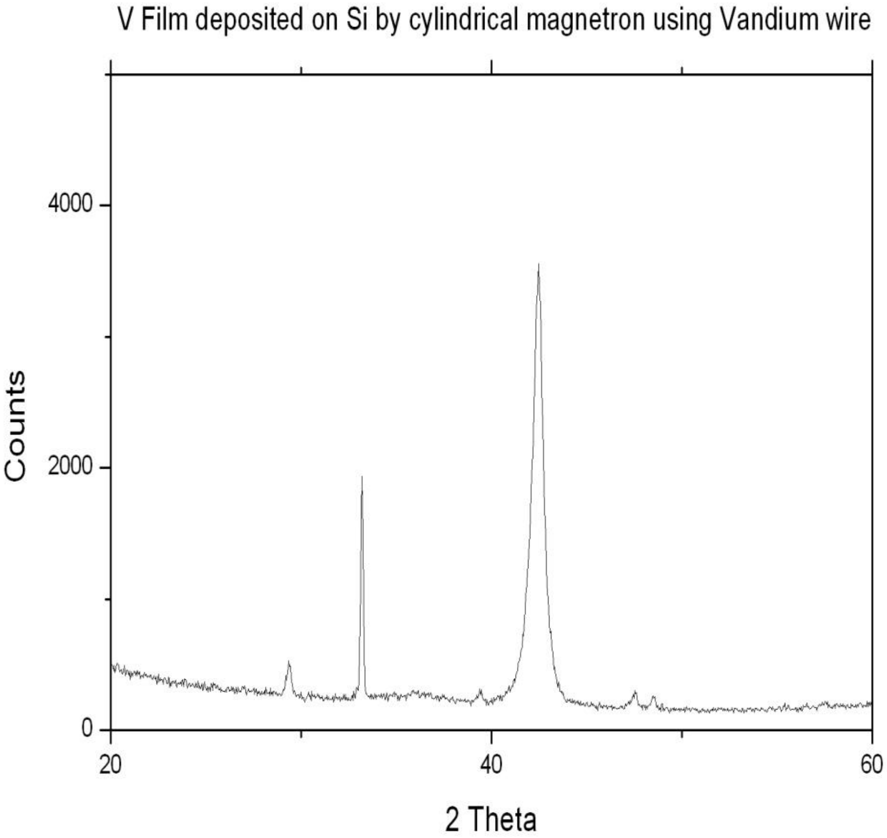
Titanium film deposited on Si test sample from a single Ti wire



Cylindrical Magnetron:
Power = 60 W,
P_{Kr} = 10^{-2} mbar,
deposition rate = 0.14 nm/s,
T = 120°C.
Average grain size 100 – 150 nm.

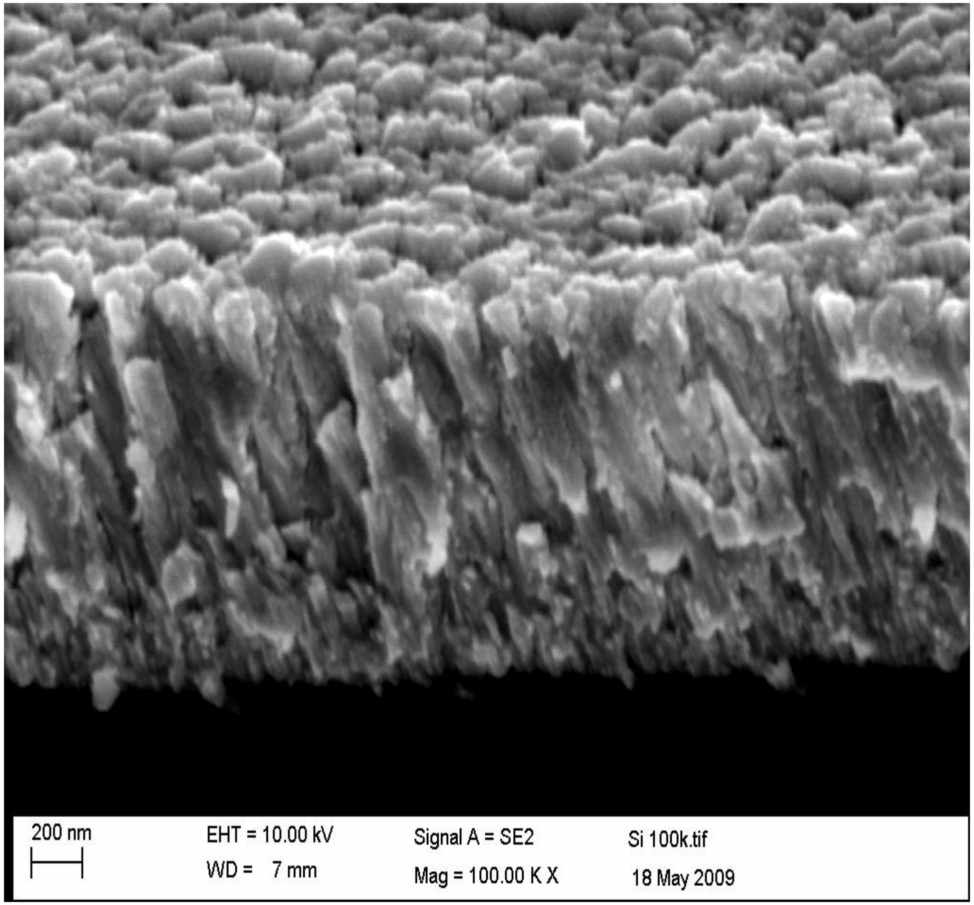
Vanadium film deposited on Si test sample from a single V wire.

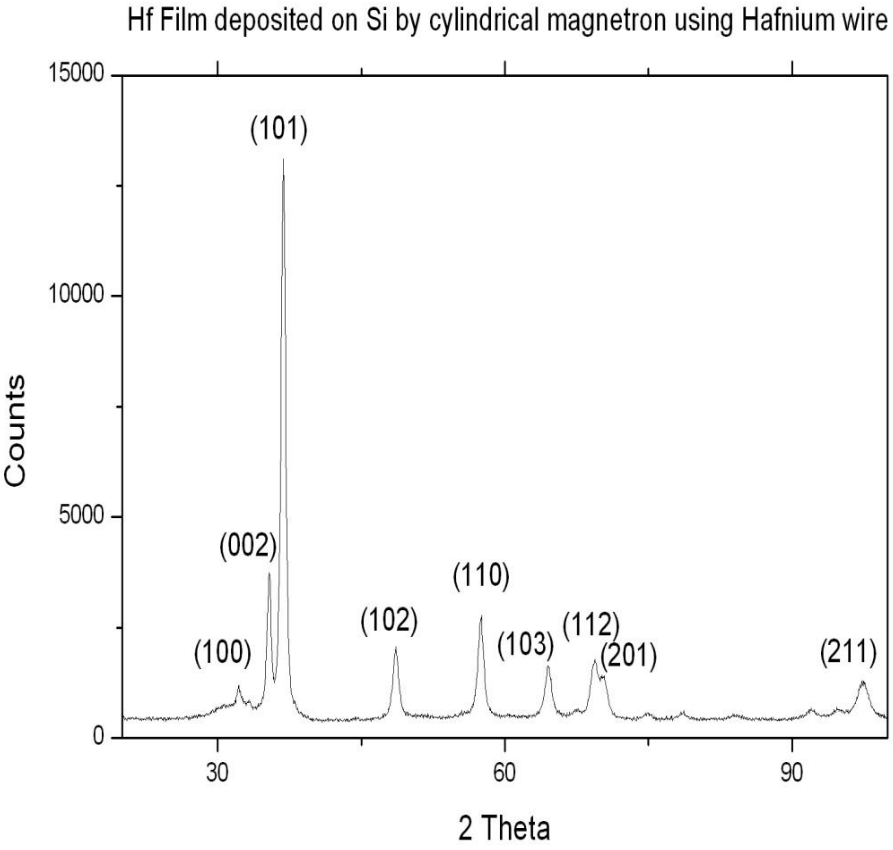




Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.16 nm/s, T = 120°C. Average grain size 100 nm. Rhombohedral lattice structure.

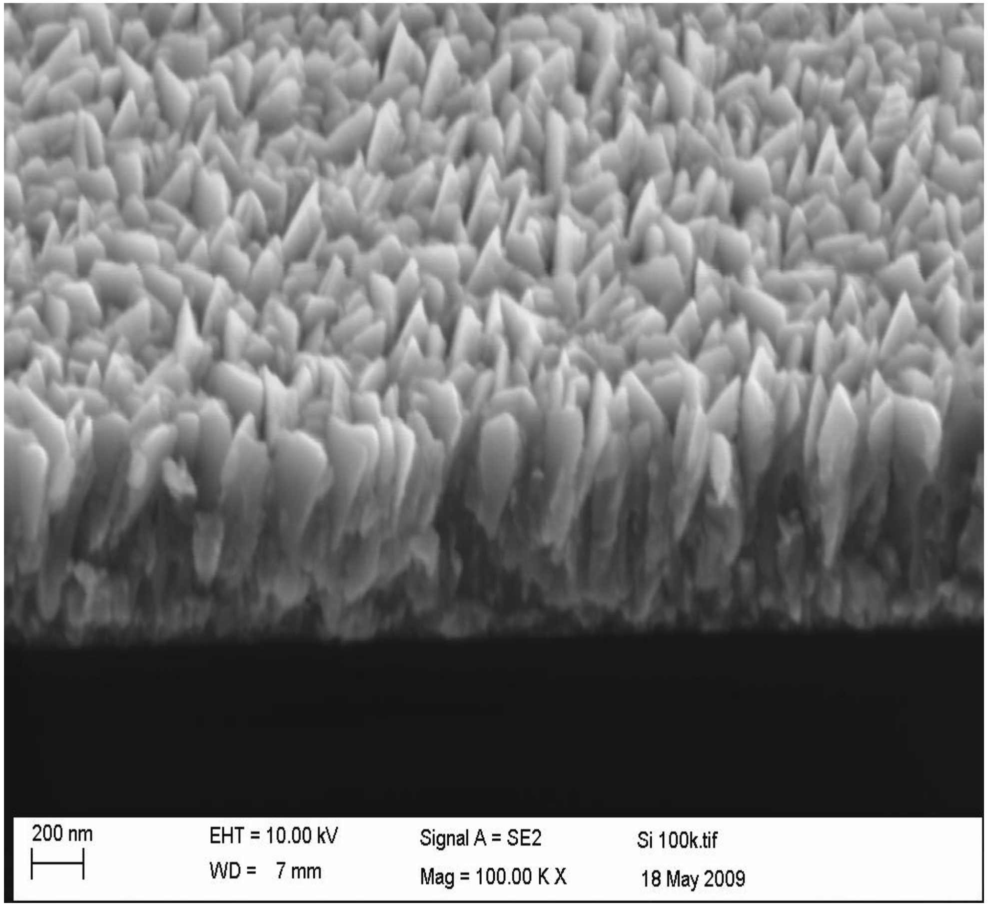
Hf film deposited on Si test sample from a single Hf wire.

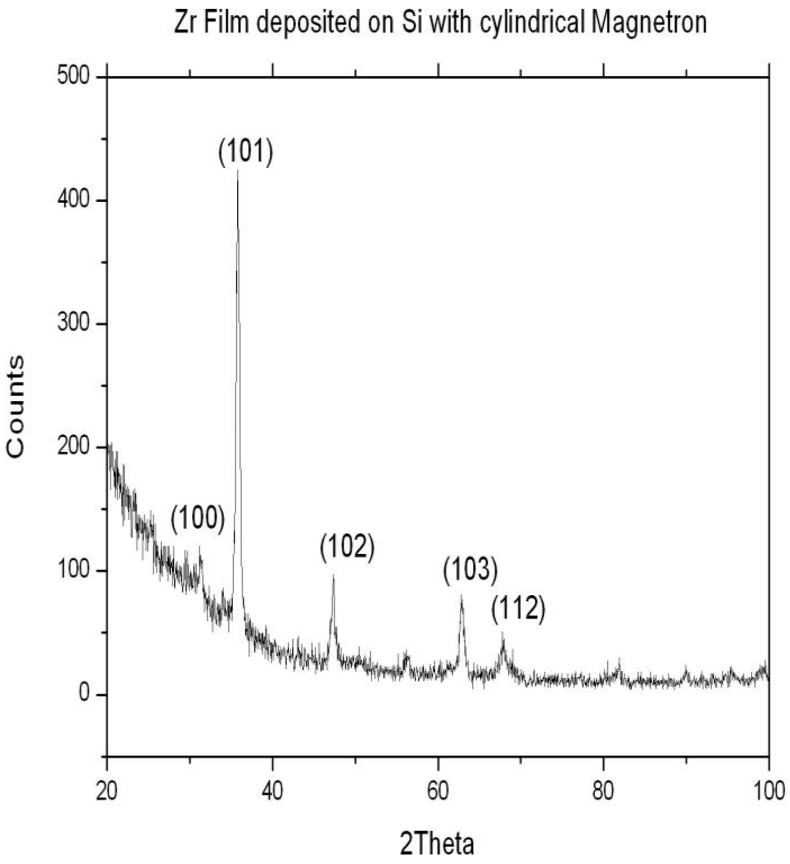




Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.16 nm/s, T = 120°C.
Average grain size 100 – 150 nm. Hexagonal lattice structure.

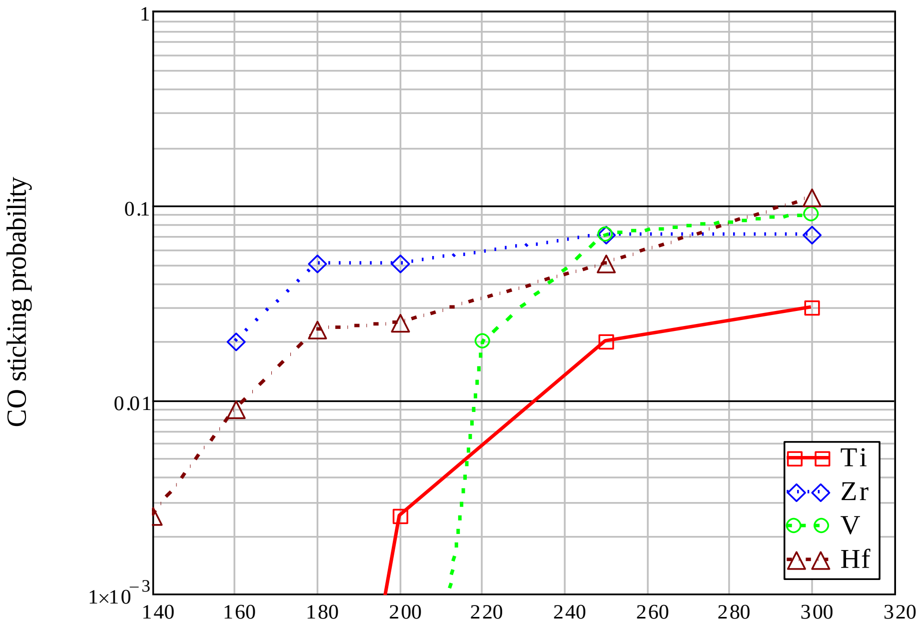
Zr film deposited on a Si test sample from a single Zr wire

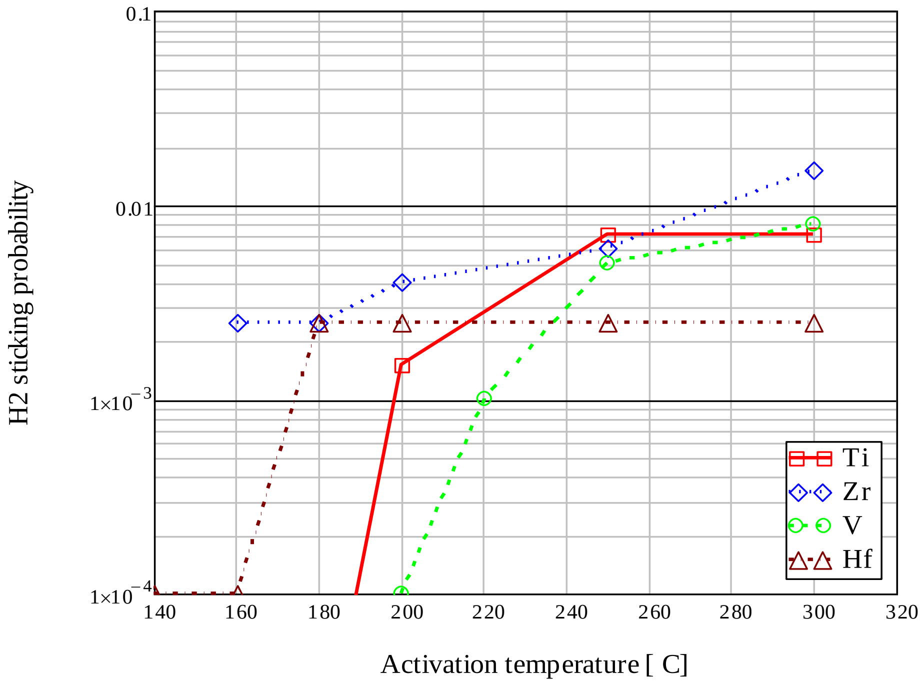


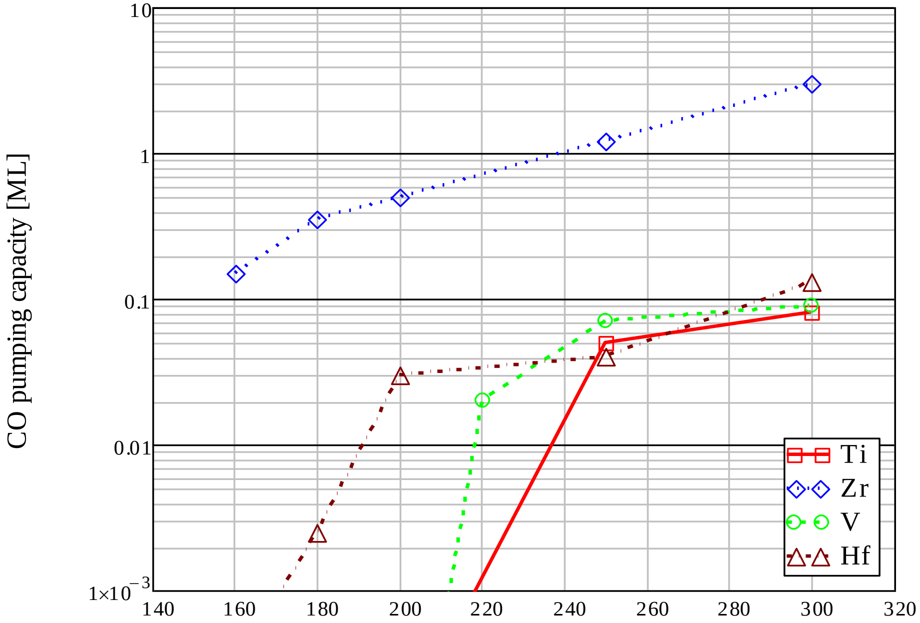

Zr Film deposited on Si with cylindrical Magnetron

Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.14 nm/s, T = 120°C. Average grain size 100 – 150 nm. Hexagonal lattice structure.

Single metal pumping properties

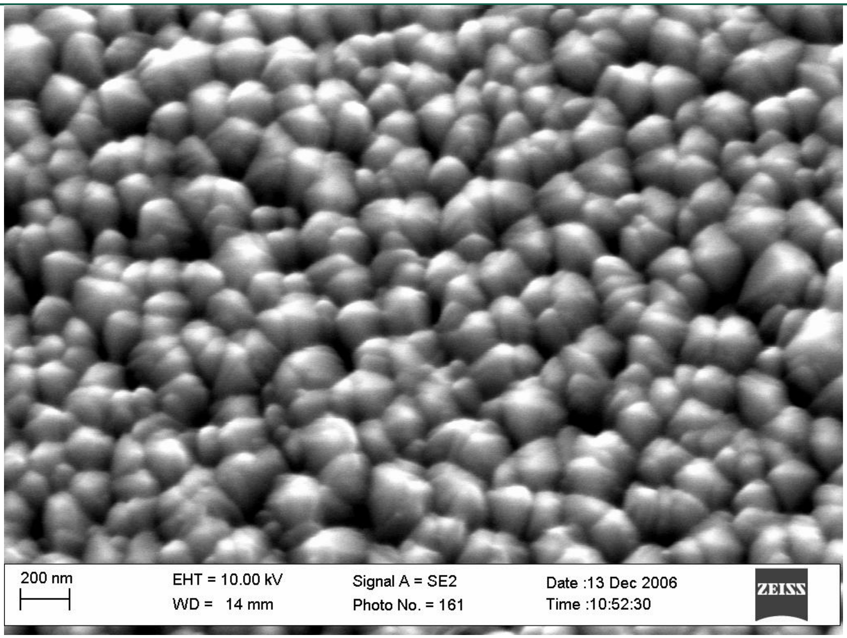


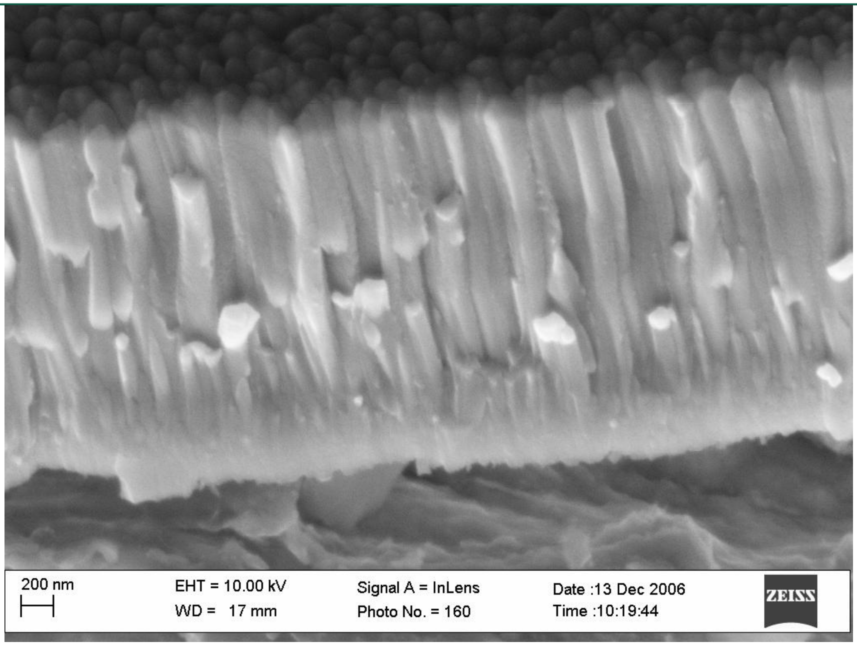


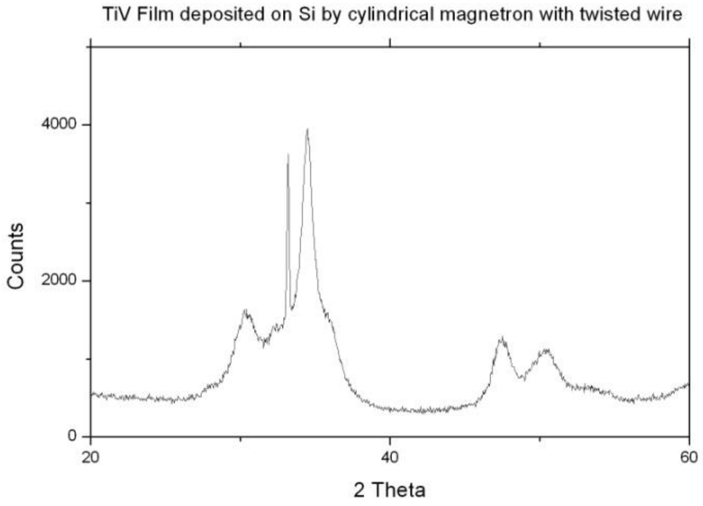


Zr is best:
Lowest activation Temp. and highest capacity
Hf
Ti
V has highest activation temperature

Ti-V film deposited on Si test sample from twisted Ti an V wires.

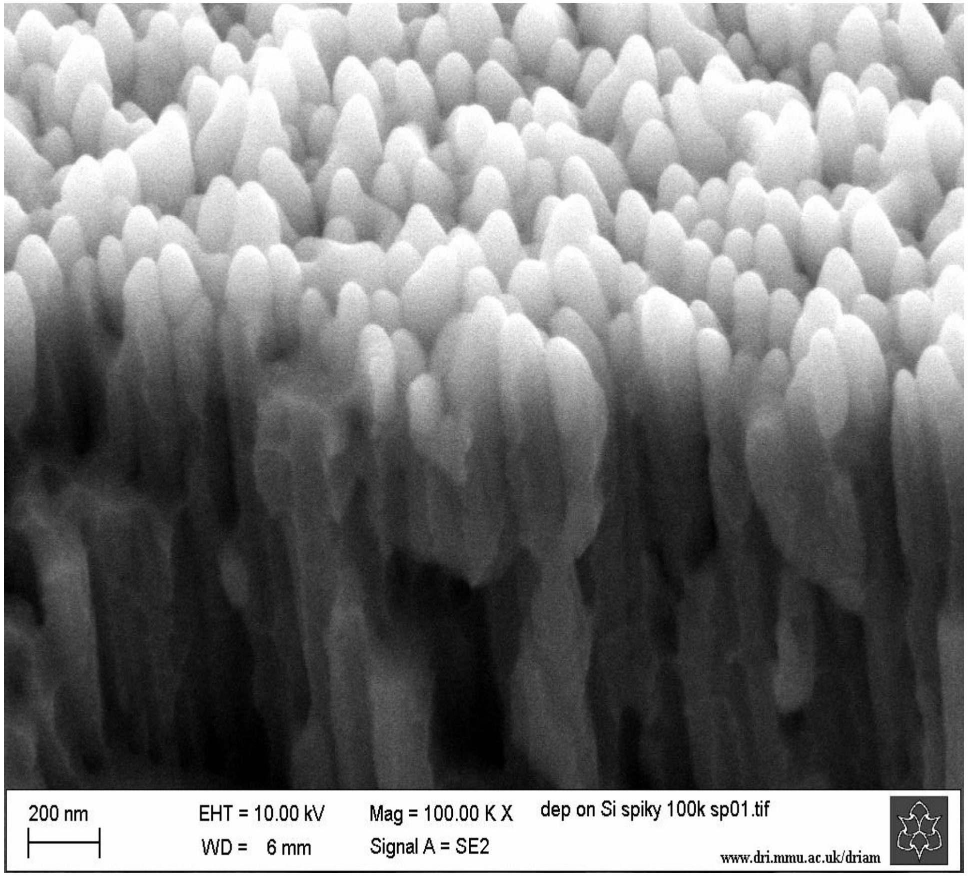


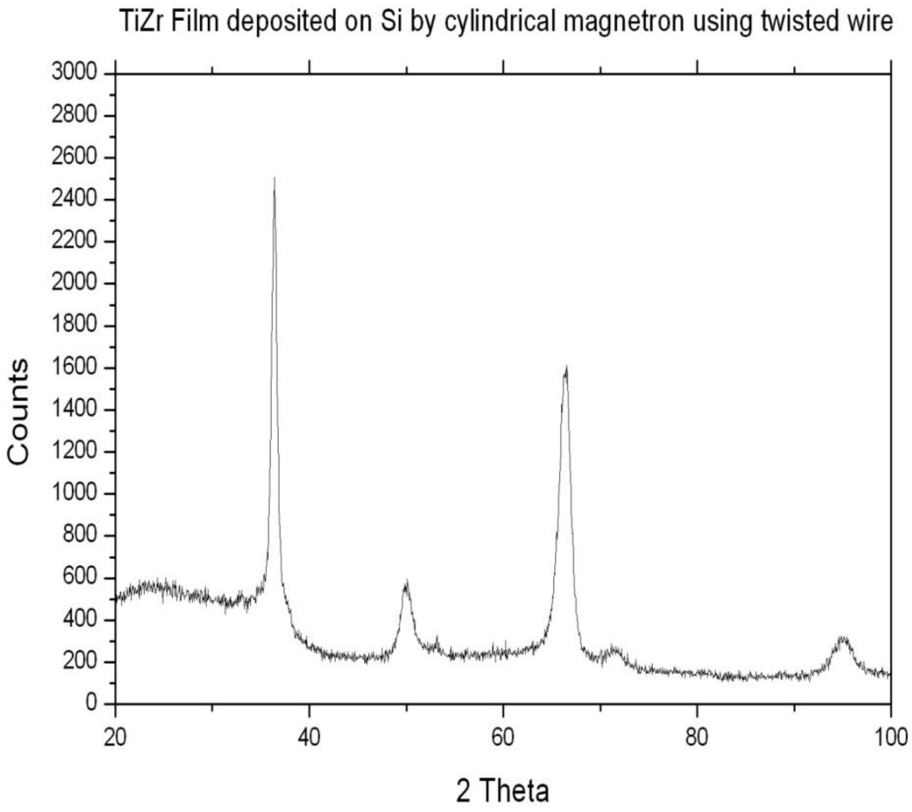




Cylindrical Magnetron:
Power = 60 W, $P_{Kr} = 10^{-2}$ mbar,
deposition rate = 0.13 nm/s, T = 120°C.
Average grain size 50 – 100 nm.
Hexagonal lattice structure.

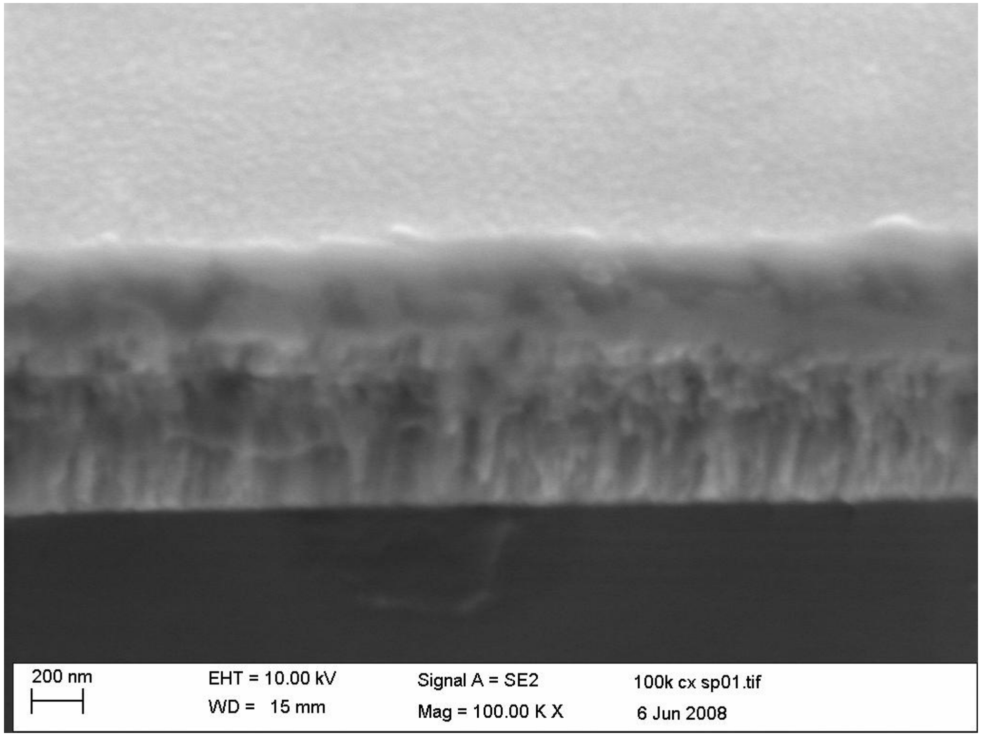
Ti-Zr film deposited on Si test sample from twisted Ti an Zr wires

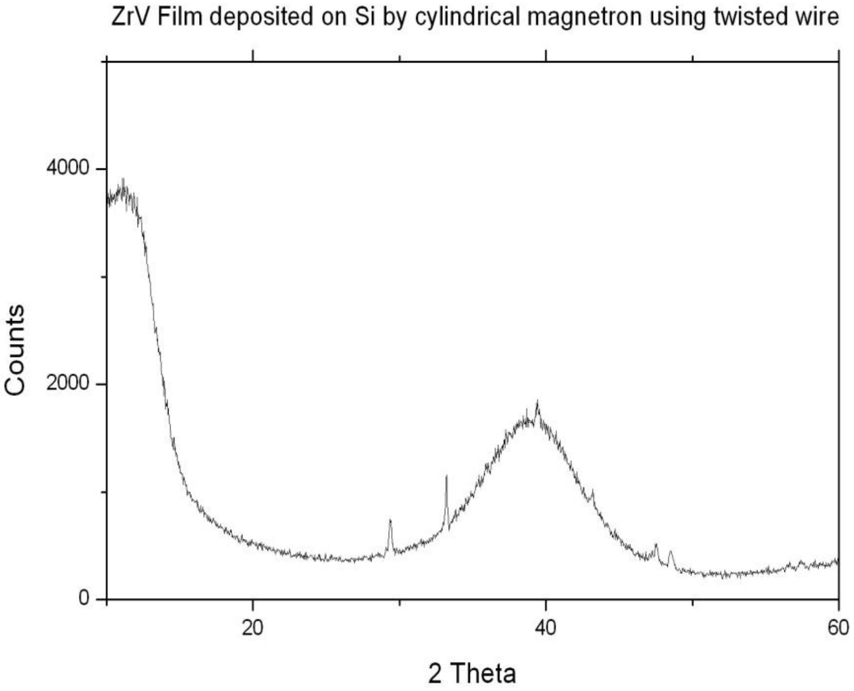



TiZr Film deposited on Si by cylindrical magnetron using twisted wire

Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.16 nm/s, T = 120°C. Average grain size 50 – 100 nm. Hexagonal lattice structure.

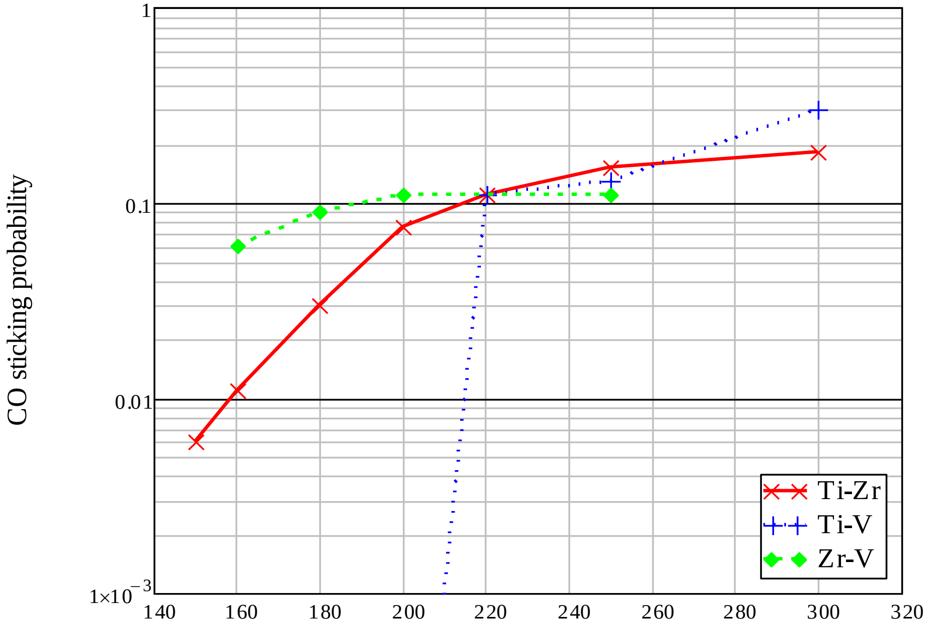
Zr-V film deposited on Si test sample from twisted Zr an V wires.

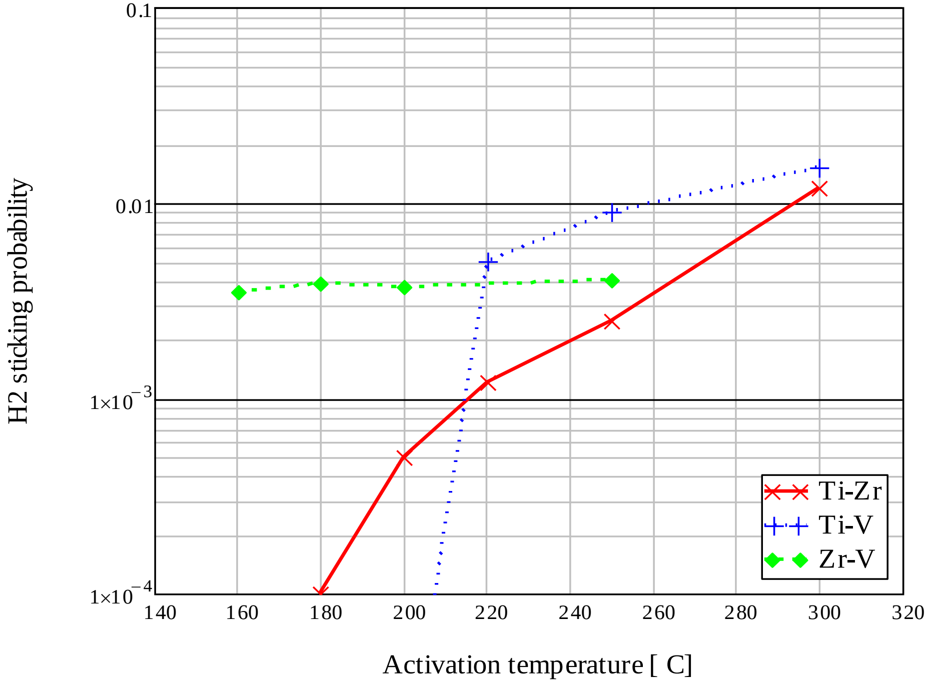


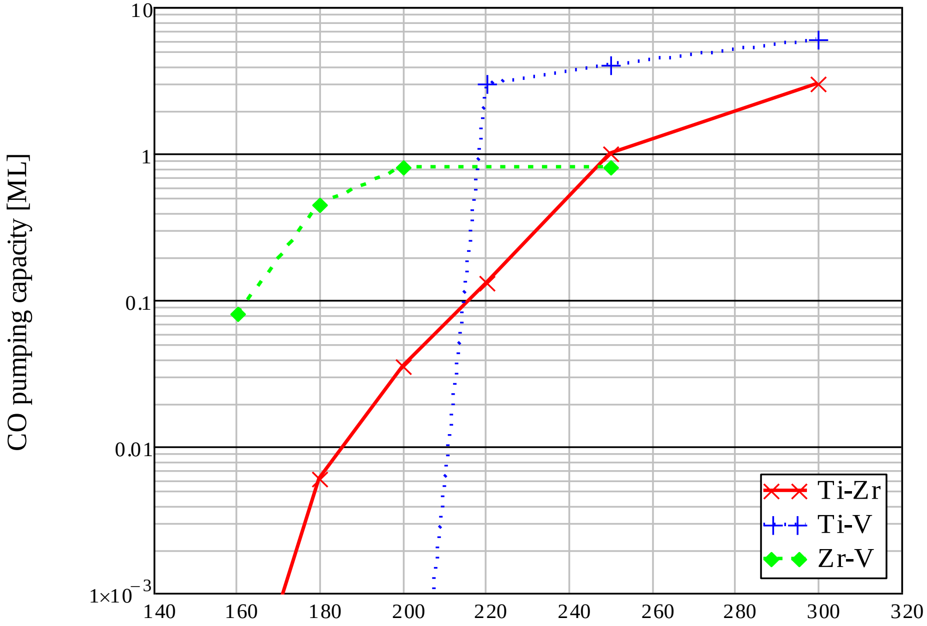


Cylindrical Magnetron: Power = 60 W, P_{Kr} = 10^{-2} mbar, deposition rate = 0.15 nm/s, T = 120°C.
Average grain size **10 – 20 nm**.

Binary alloy pumping properties





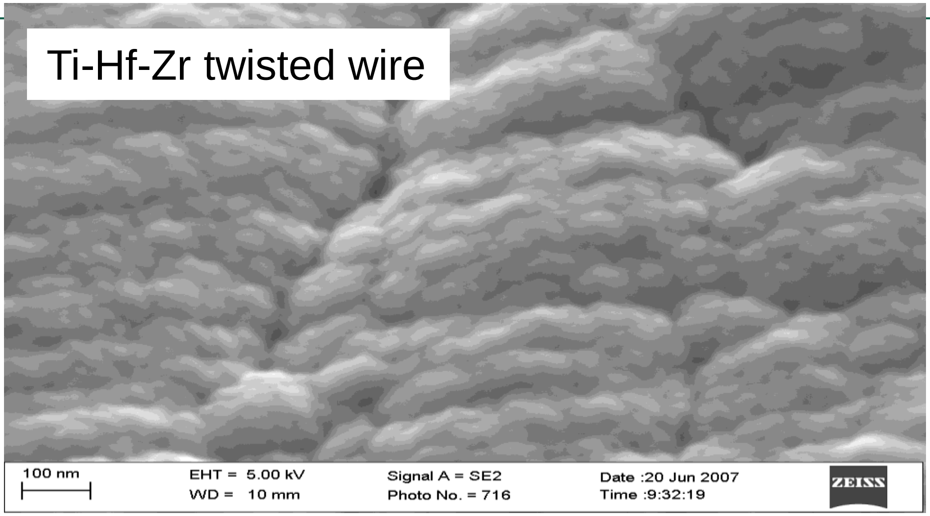


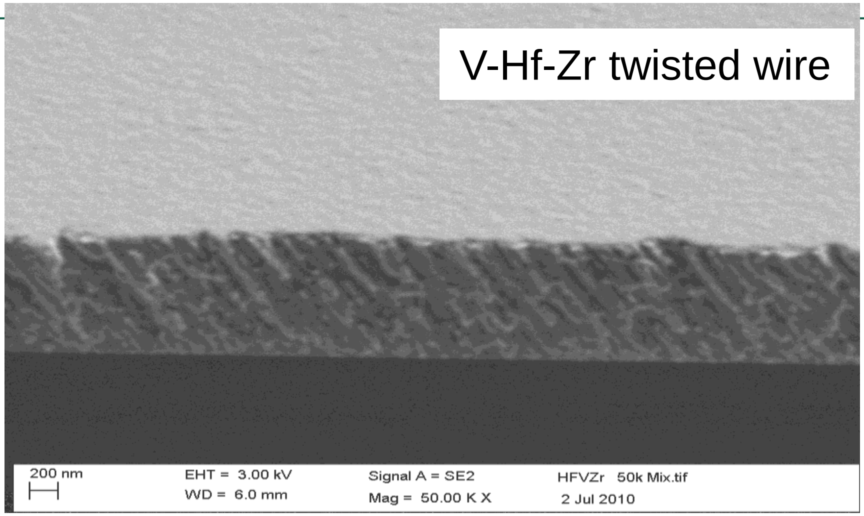
Zr-V is best

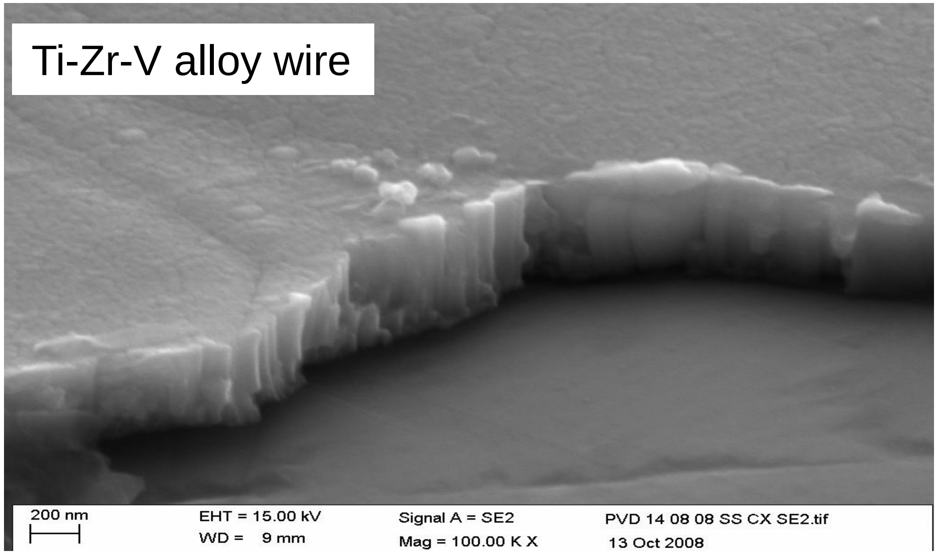
Ti-Zr activation temperature is lower than for **Ti-V**

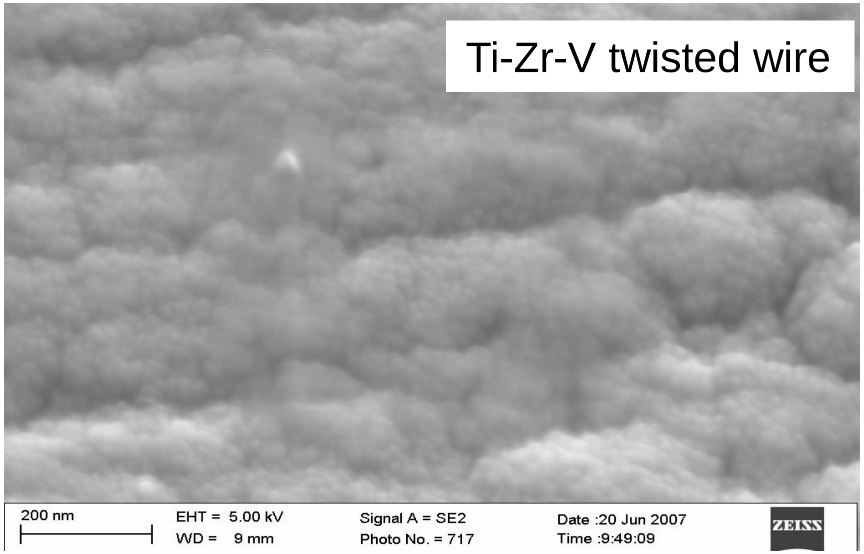
Zr-Hf was not studied

Ternary NEG film deposited on Si test sample from twisted Ti, V, Zr, and Hf wires and TiZrV alloy wire


Ti-Hf-Zr twisted wire

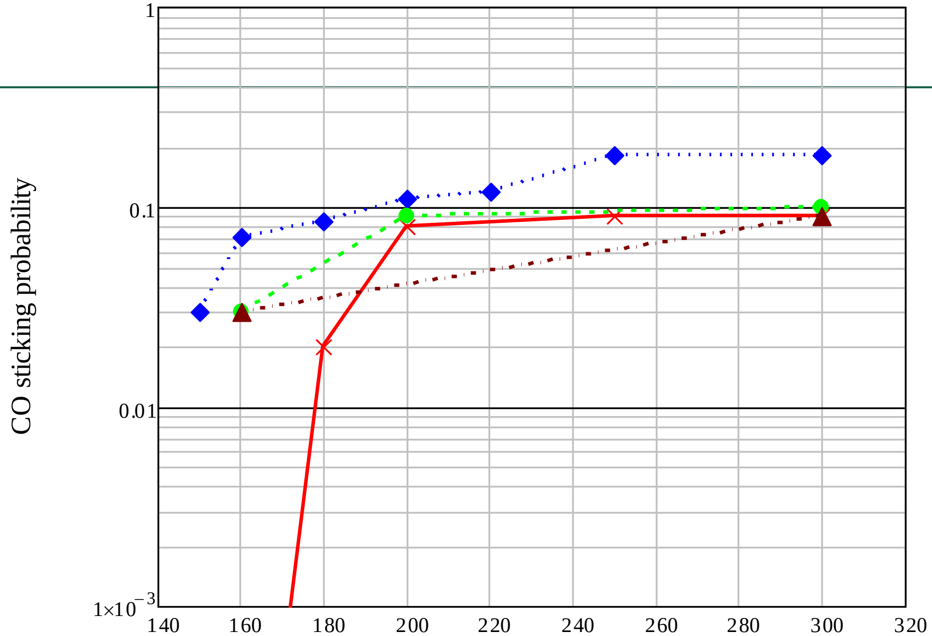

V-Hf-Zr twisted wire

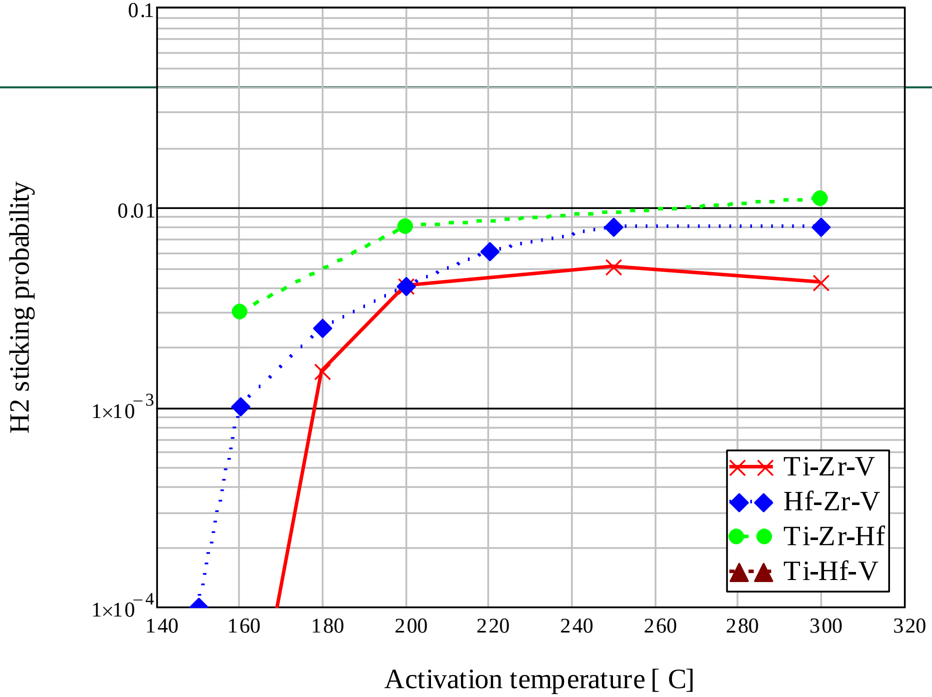

Ti-Zr-V alloy wire

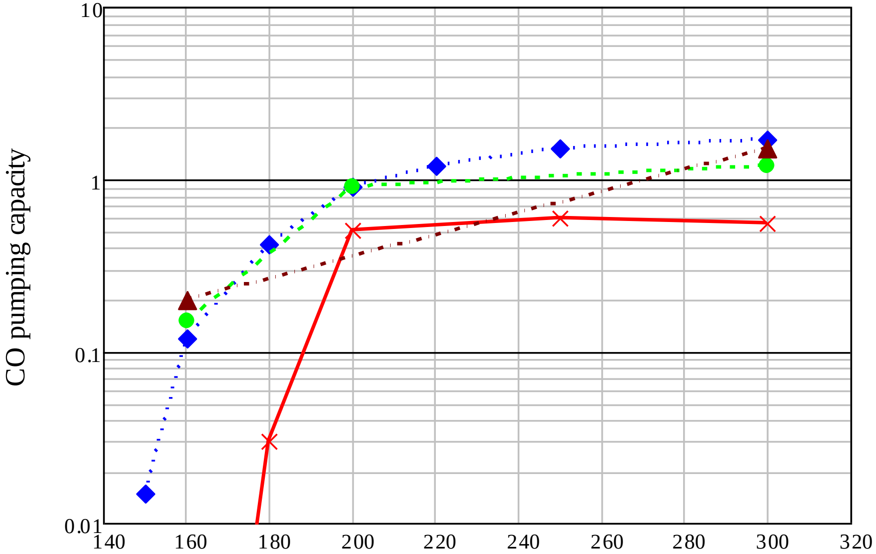

Ti-Zr-V twisted wire

Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.12 nm/s, T = 120°C. Average grain size 5 nm. Hexagonal lattice structure.

Ternary alloy pumping properties



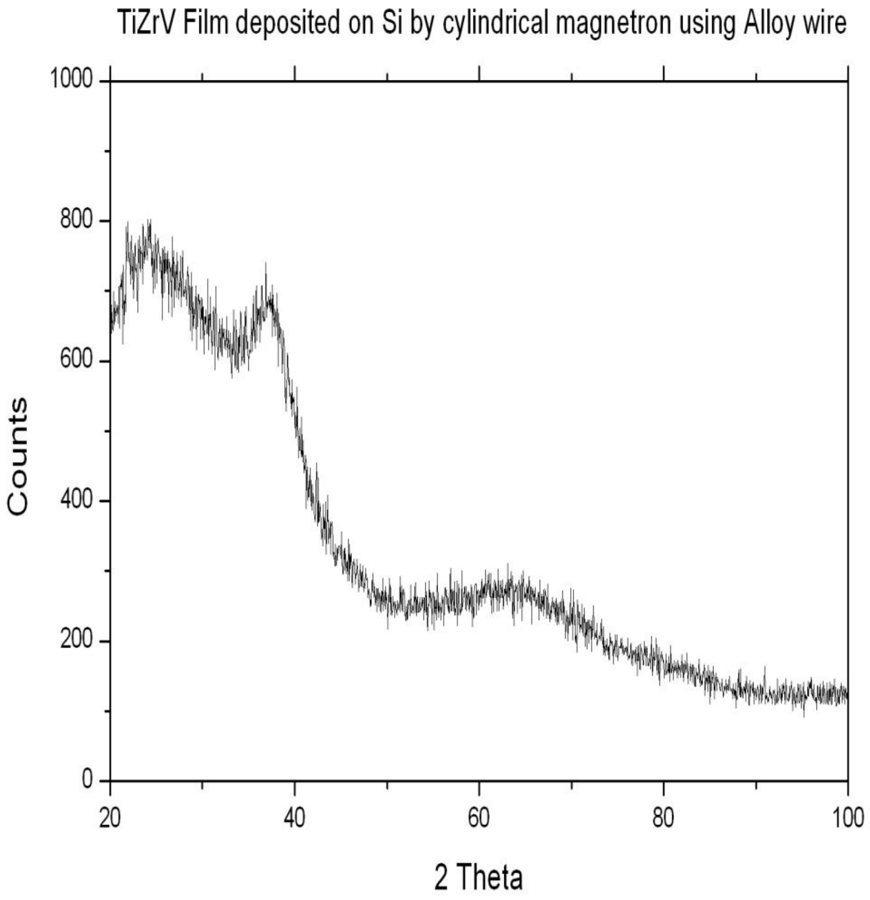


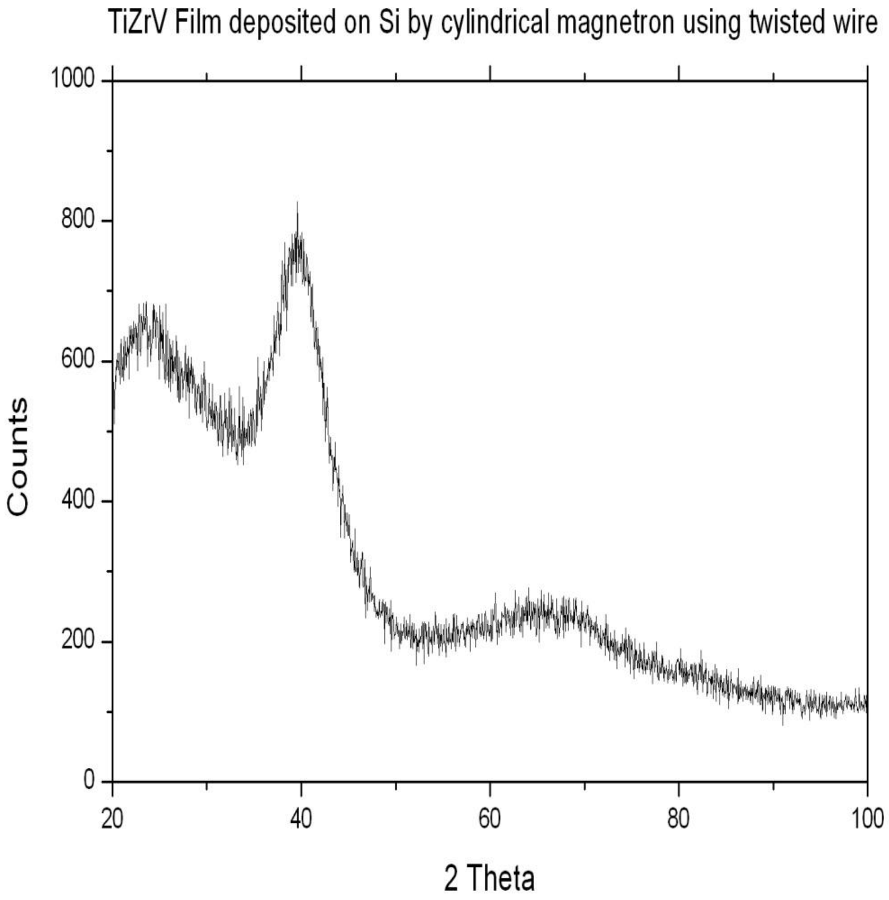


Hf-Zr-V, Ti-Zr-Hf and **Ti-Hf-V** are comparable

Ti-Zr-V has the highest activation temperature

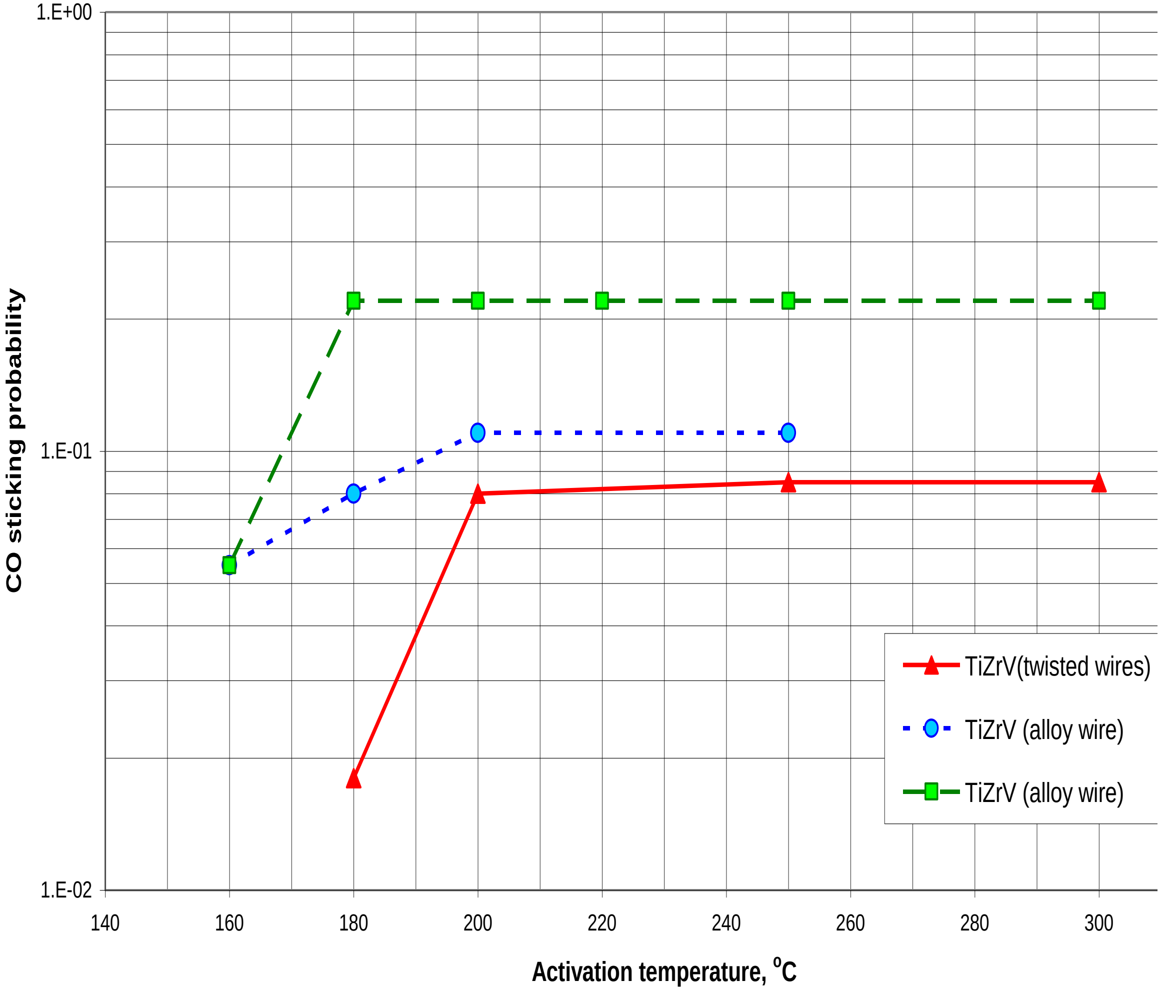
XRD of Ti-Zr-V film: alloy wire vs. twisted wires as target.

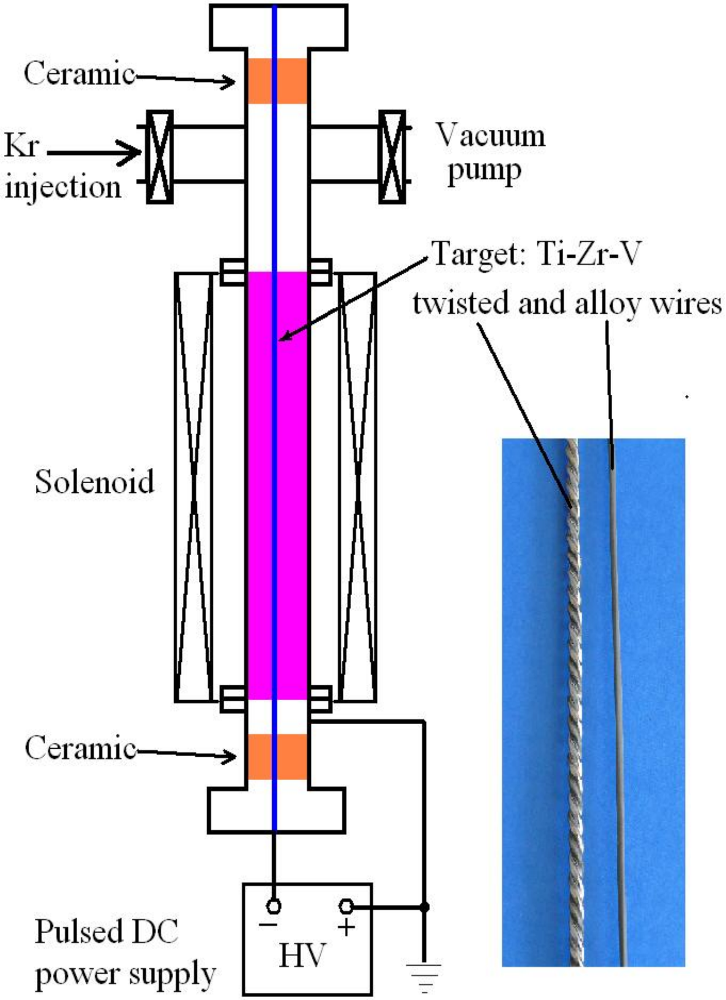




In Both cases there is only one broad peak near $2\theta = 40°$
The film is nearly amorphous.

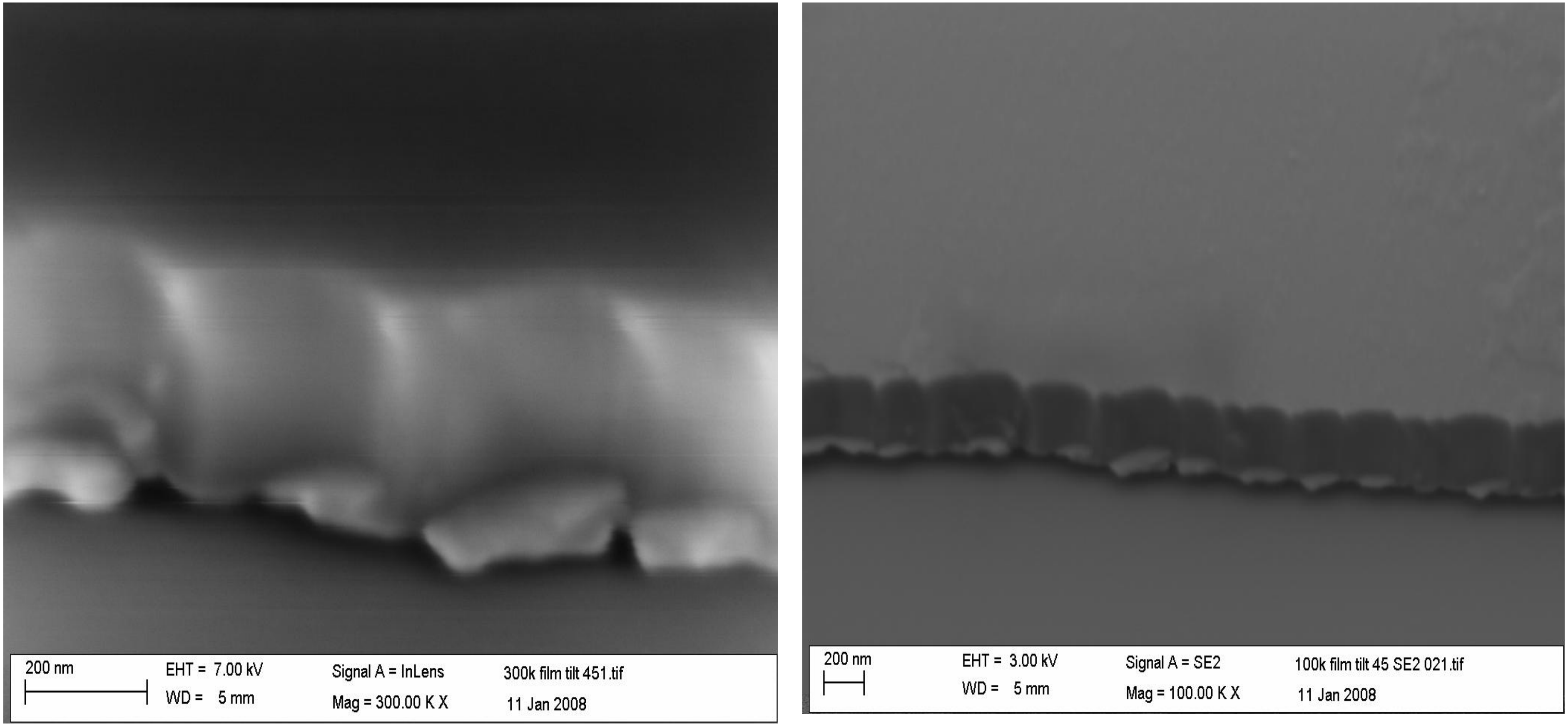
Twisted wires vs. alloy target: reducing Ta





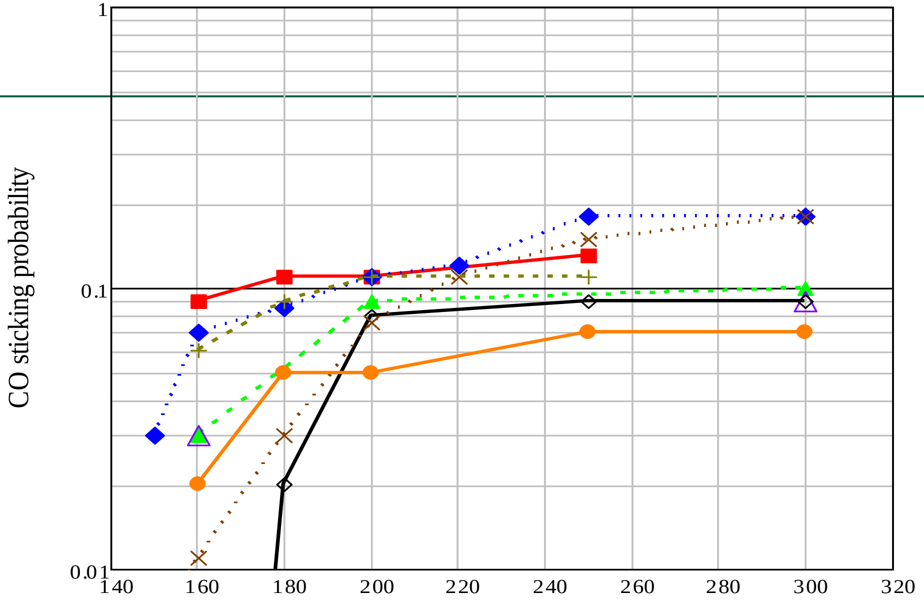
R. Valizadeh, O.B. Malyshev, J.S. Colligon, V. Vishnyakov. Accepted by J. Vac. Sci. Technol. Aug. 2010.

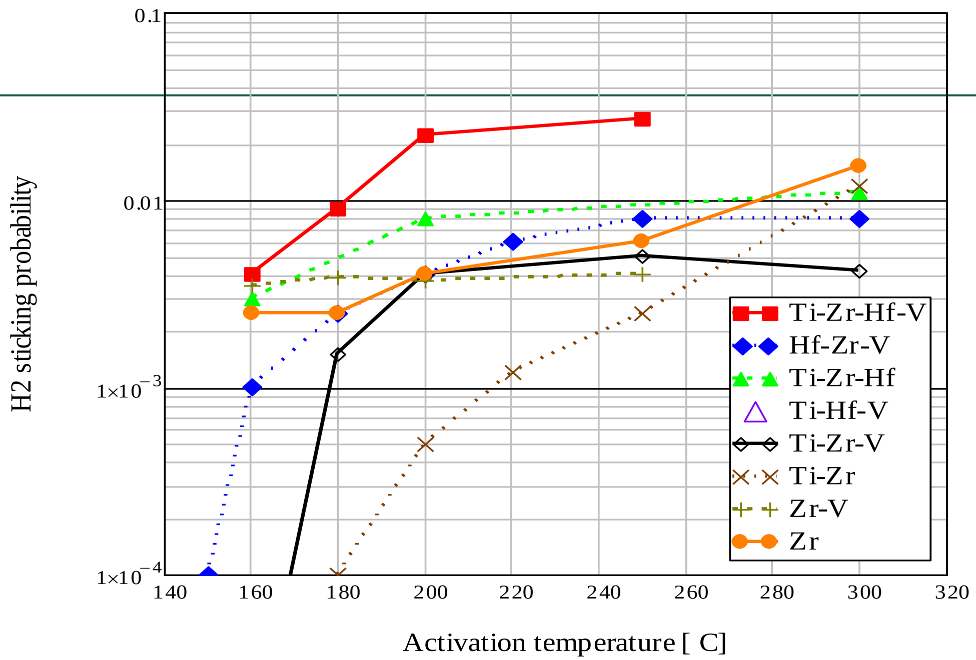
Quaternary NEG alloy film deposited on Si test sample from twisted Ti, V, Zr, and Hf wires.

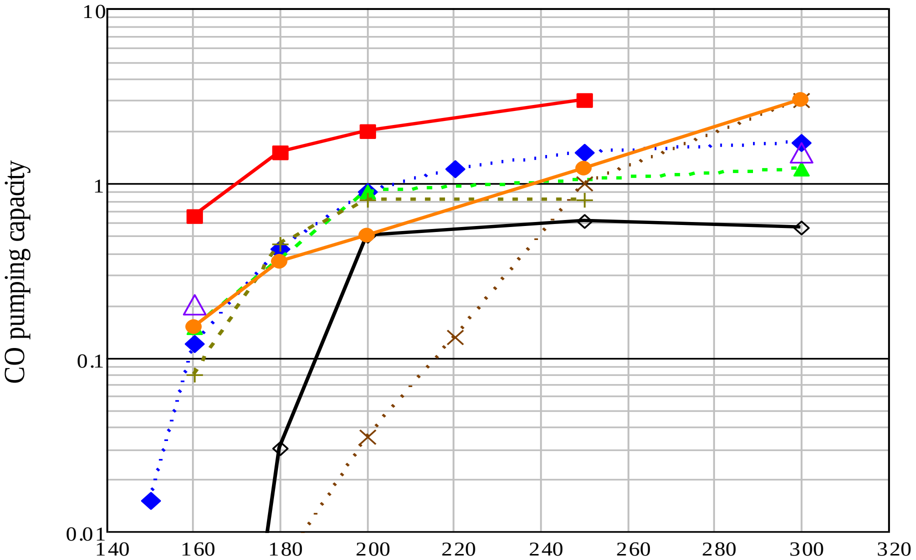


Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.12 nm/s, T = 120°C.
Very glassy structure.

Quaternary alloy pumping properties







Ti-Zr-Hf-V is the best

Hf-Zr-V, **Ti-Zr-Hf**, **Ti-Hf-V** and **Zr** are comparable

Ti-Zr-V is lower

Zr-V (best binary alloy) has the lowest activation temperature

Pressure in the accelerator vacuum chamber

$$P \propto \frac{\eta}{\alpha}$$

where

- η - desorption yield
- α - sticking probability

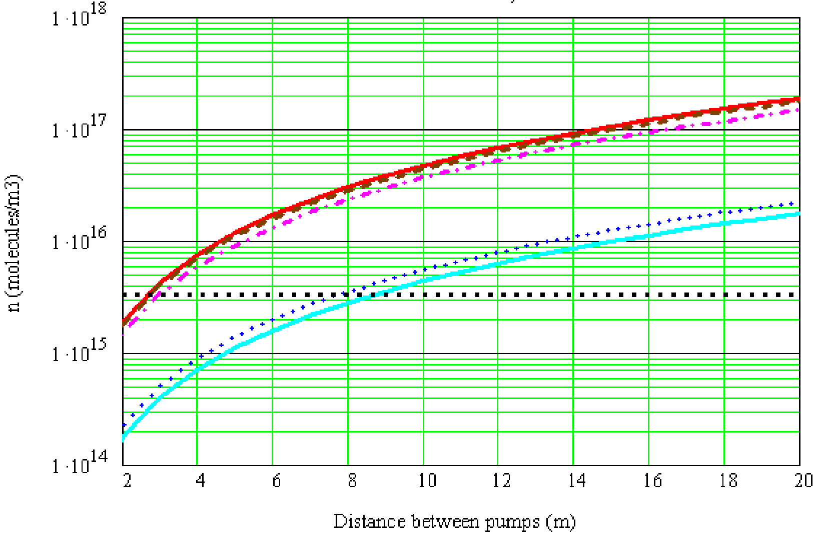
- Improving pumping properties is limited:

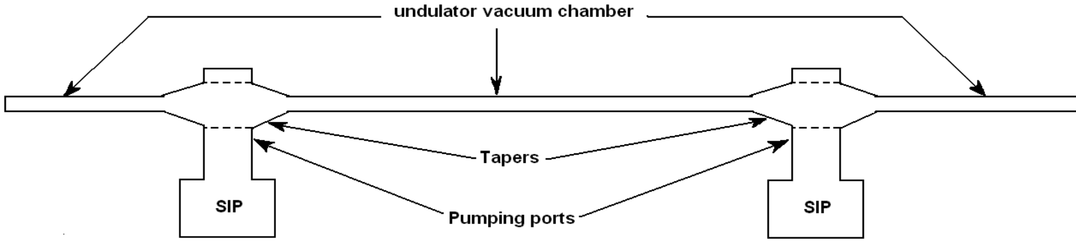
  $\alpha \leq \mathbf{1}$.

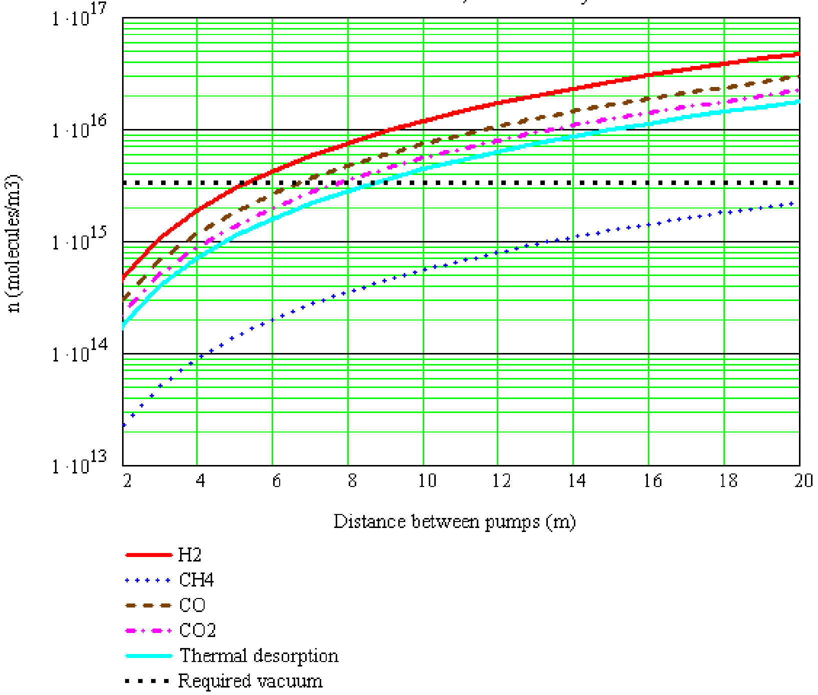
  - $0.005 < \alpha_{H2} < 0.01$
  - $0.1 < \alpha_{CO} < 0.5$
  - $0.4 < \alpha_{CO2} < 0.6$
- Reducing the desorption yields η

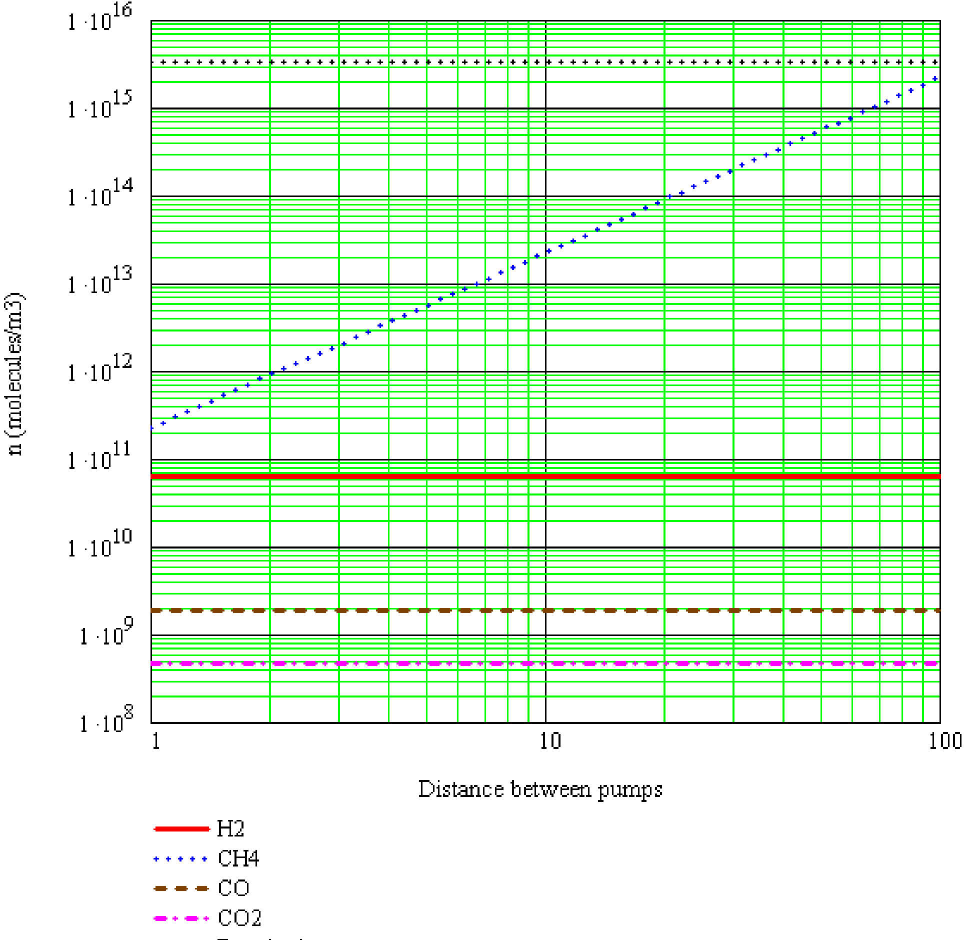
. <u>in orders of magnitude</u> is a realistic task

Average gas density in the ILC undulator: d=4 mm


Stainless steel tube, initial




Stainless steel tube, after 100 days

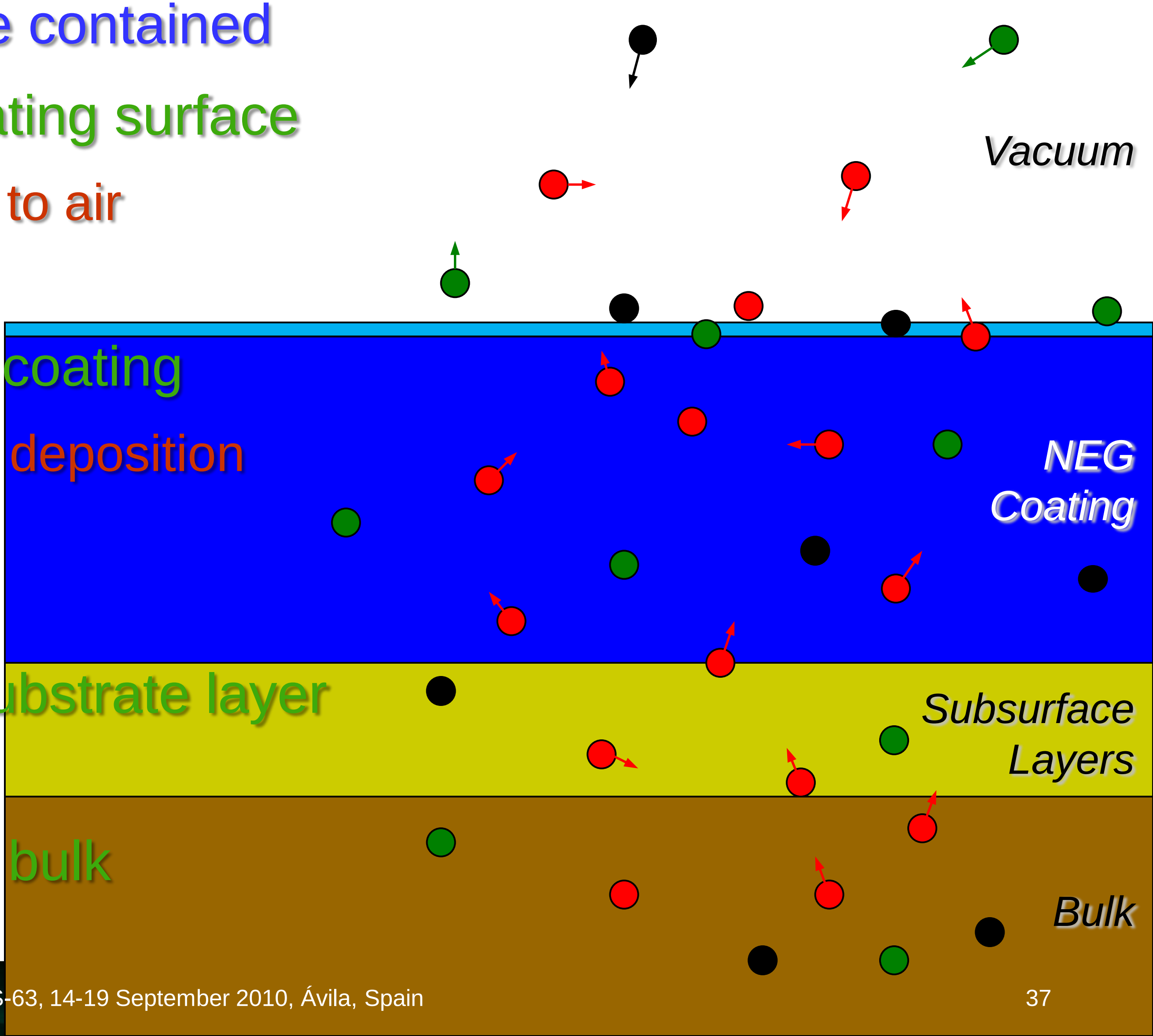

NEG coated tube

Reducing the gas desorption from the NEG coatings

- Main gases in the NEG coated vacuum chamber are H_2 and CH_4
  - Only H_2 can diffuse through the NEG film under bombardment or heat
  - CH_4 is most likely created on the NEG surface from diffused H_2 and C (originally from sorbed CO and CO_2)
  - Therefore the H_2 diffusion must be suppressed
    - **Where H_2 come from?**

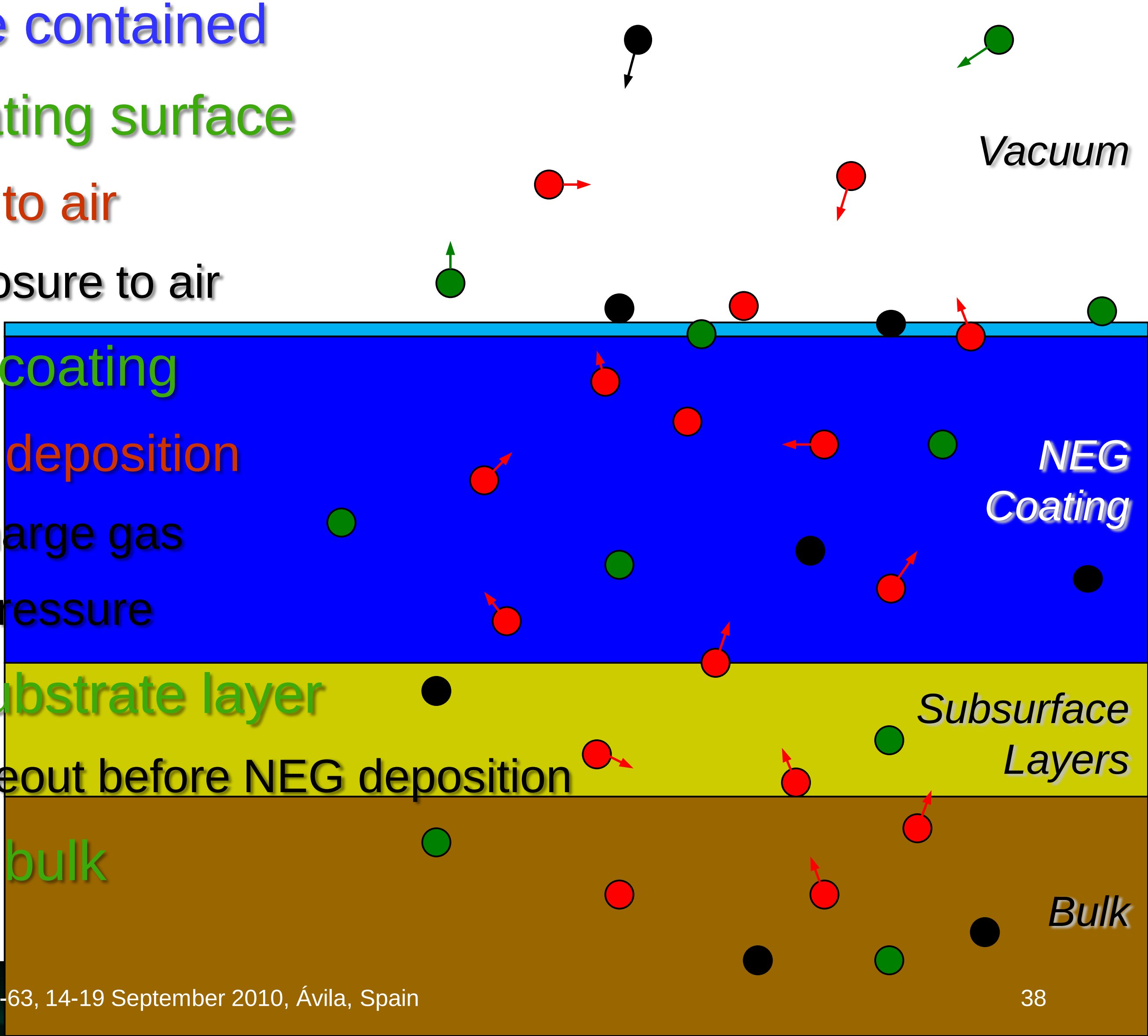
Reducing the gas desorption from the NEG coatings

- Gas molecules are contained
  - on the NEG coating surface
    - after exposure to air
  - inside the NEG coating
    - trapped during deposition
  - in subsurface substrate layer
  - in the substrate bulk



Reducing the gas desorption from the NEG coatings

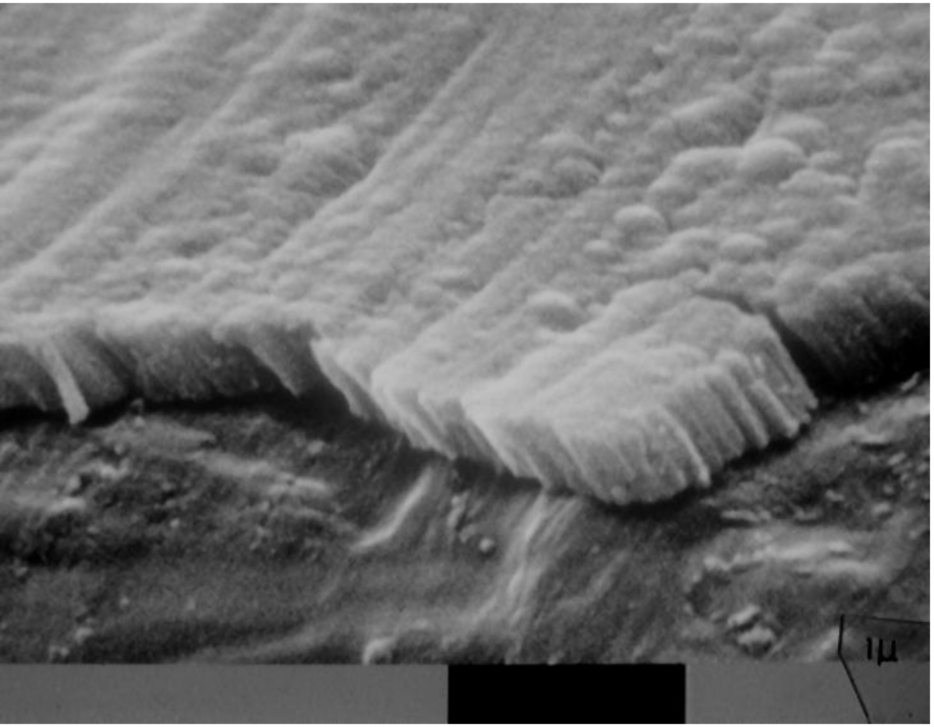
- Gas molecules are contained
  - on the NEG coating surface
    - after exposure to air
      - minimise exposure to air
  - inside the NEG coating
    - trapped during deposition
      - purity of discharge gas
      - background pressure
  - in subsurface substrate layer
      - substrate bakeout before NEG deposition
  - in the substrate bulk
      - vacuum firing



SEM images of films (film morphology)

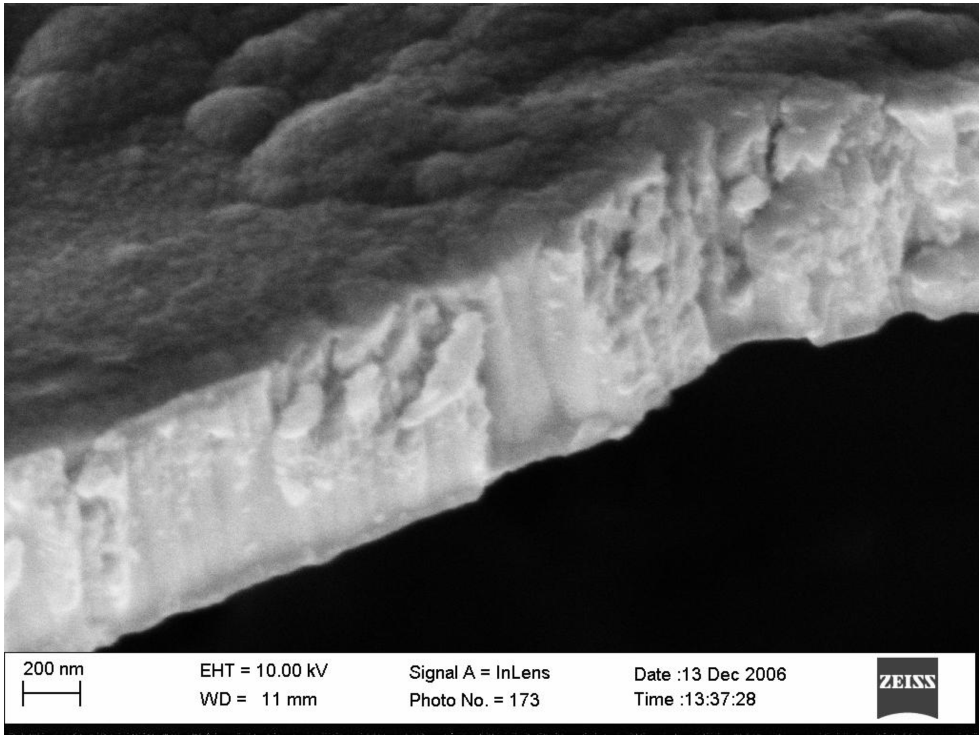
columnar

Best for pumping



dense

A first candidate for a barrier

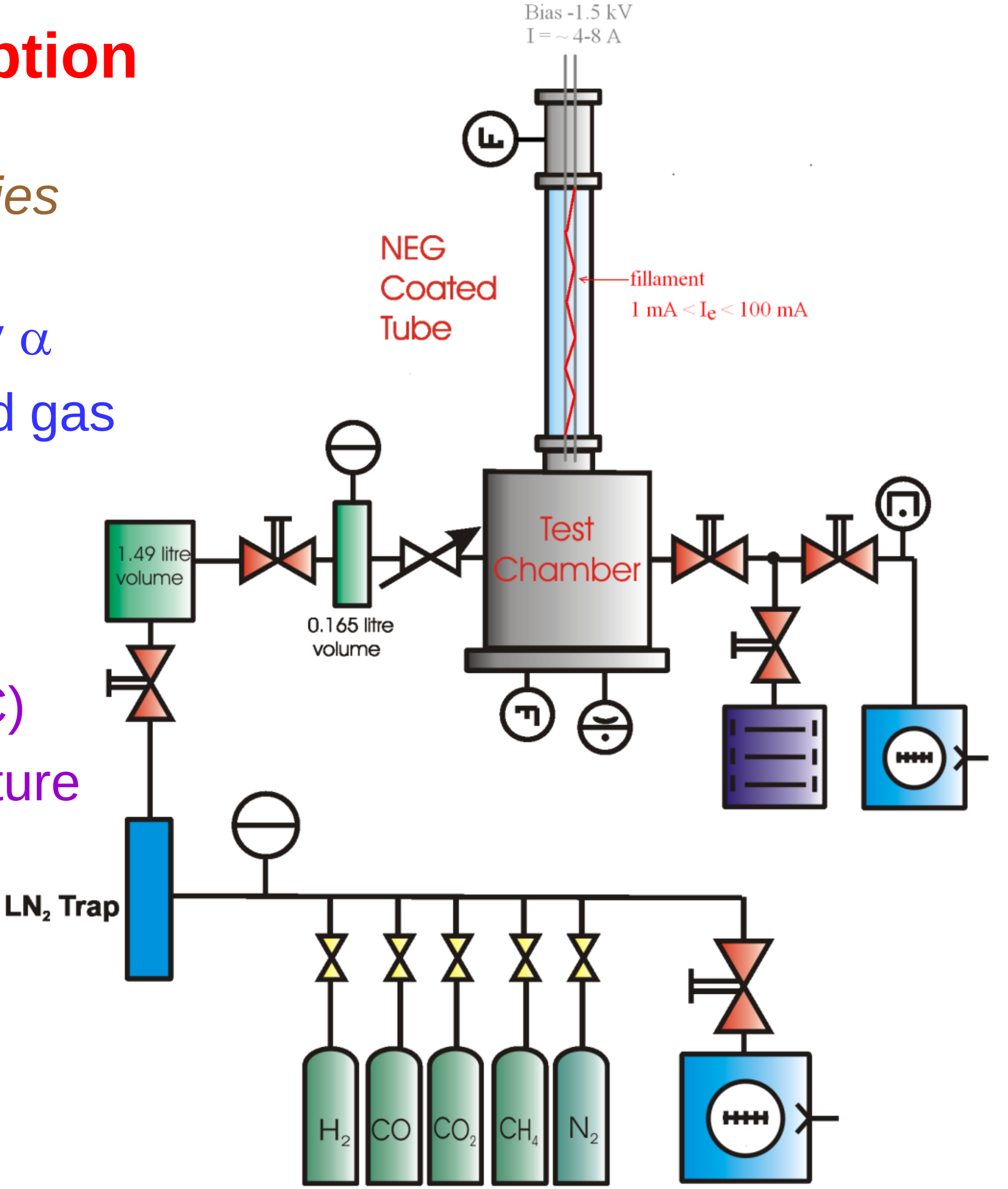


O.B. Malyshev, R. Valizadeh, J.S. Colligon *et al*. J. Vac. Sci. Technol. A 27 (2009), p. 521.

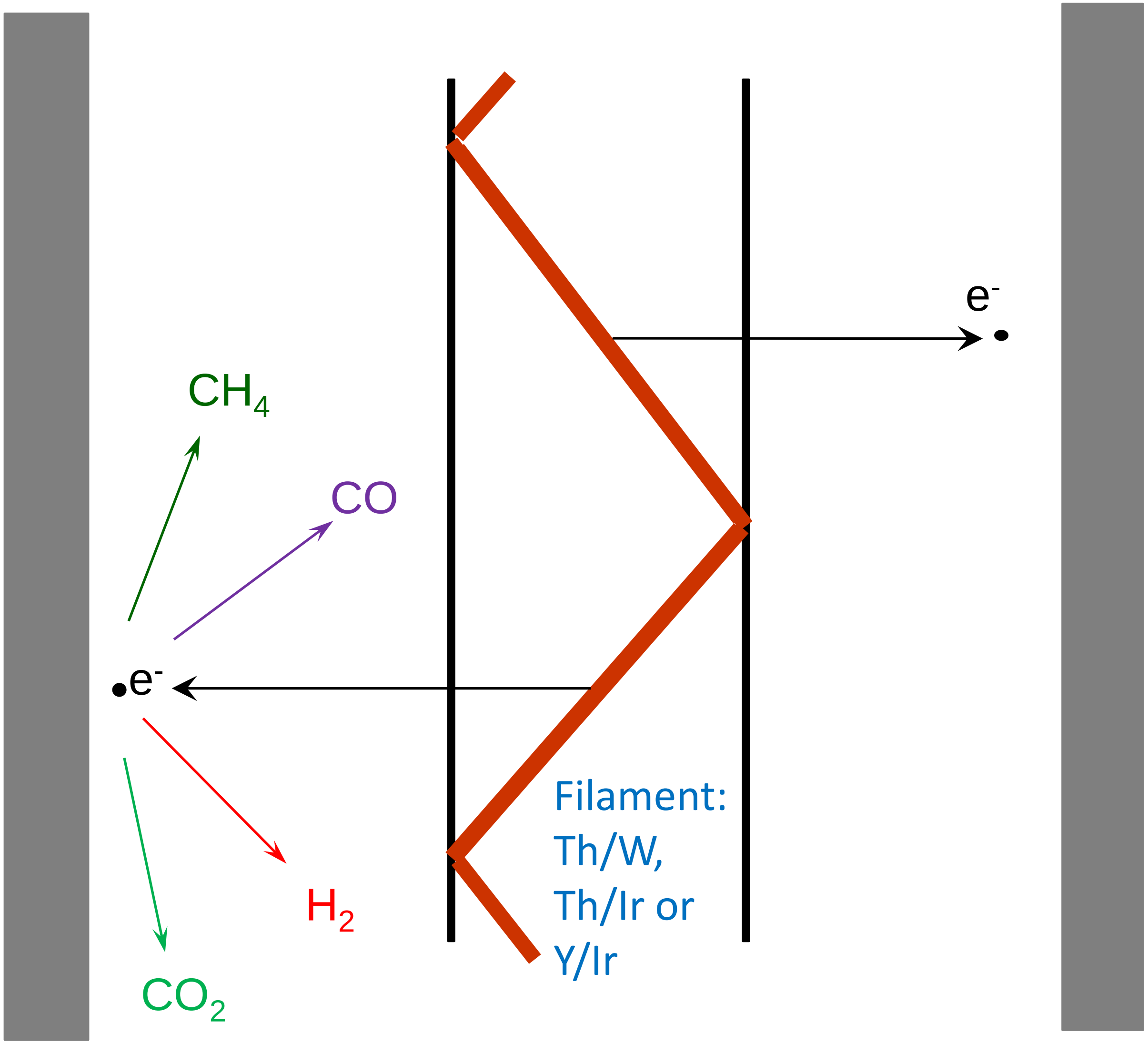
Electron stimulated desorption

Modified NEG pumping properties evaluation rig:

- To measure sticking probability α
- To measure electron stimulated gas desorption as a function of
  - Electron energy
  - Dose
  - Wall temperature (20-100°C)
  - Activation/bakeout temperature
- Can be used for samples with:
  - NEG coating
  - Low desorption coating
  - No coatings



Electron Bombardment

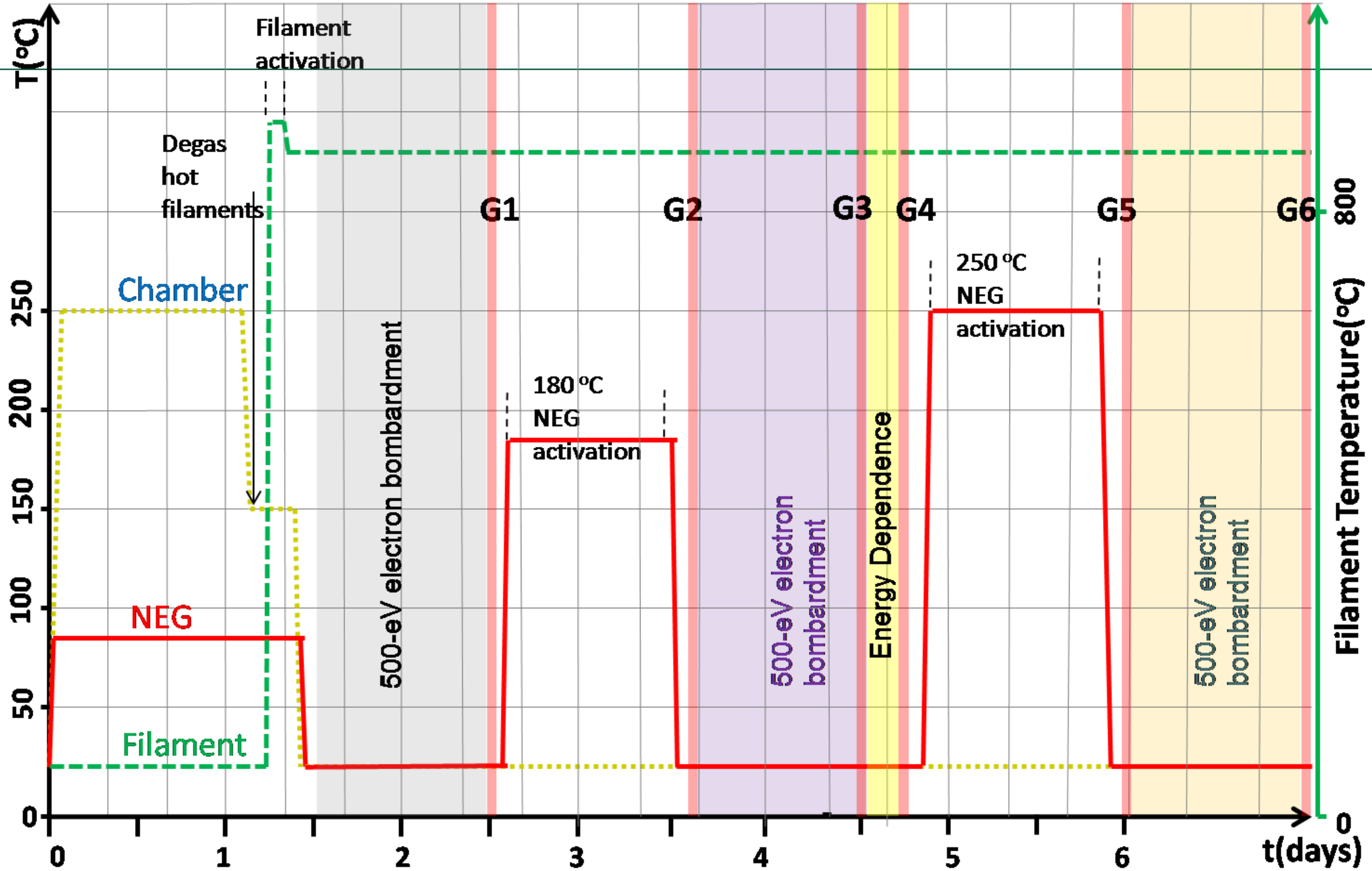


Filament: Th/W, Th/Ir or Y/Ir

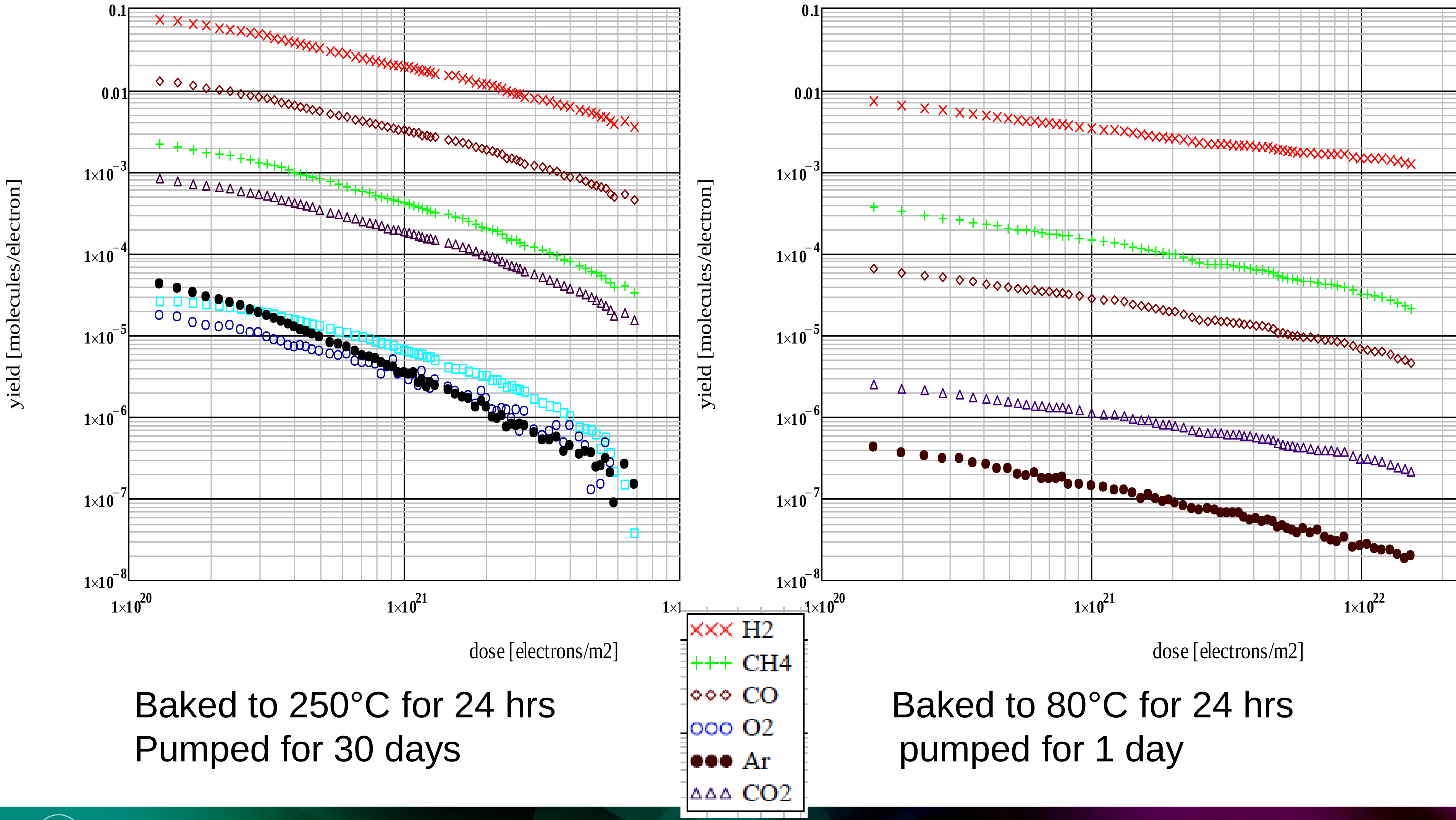
Electron Stimulated Desorption (ESD) studies programme

- ESD as a function of
  - Activation/bakeout temperature
  - Electron energy
  - Electron dose
  - Coating density, morphology and structure
  - Deposition conditions
  - Substrate

Experimental procedure for NEG coated samples



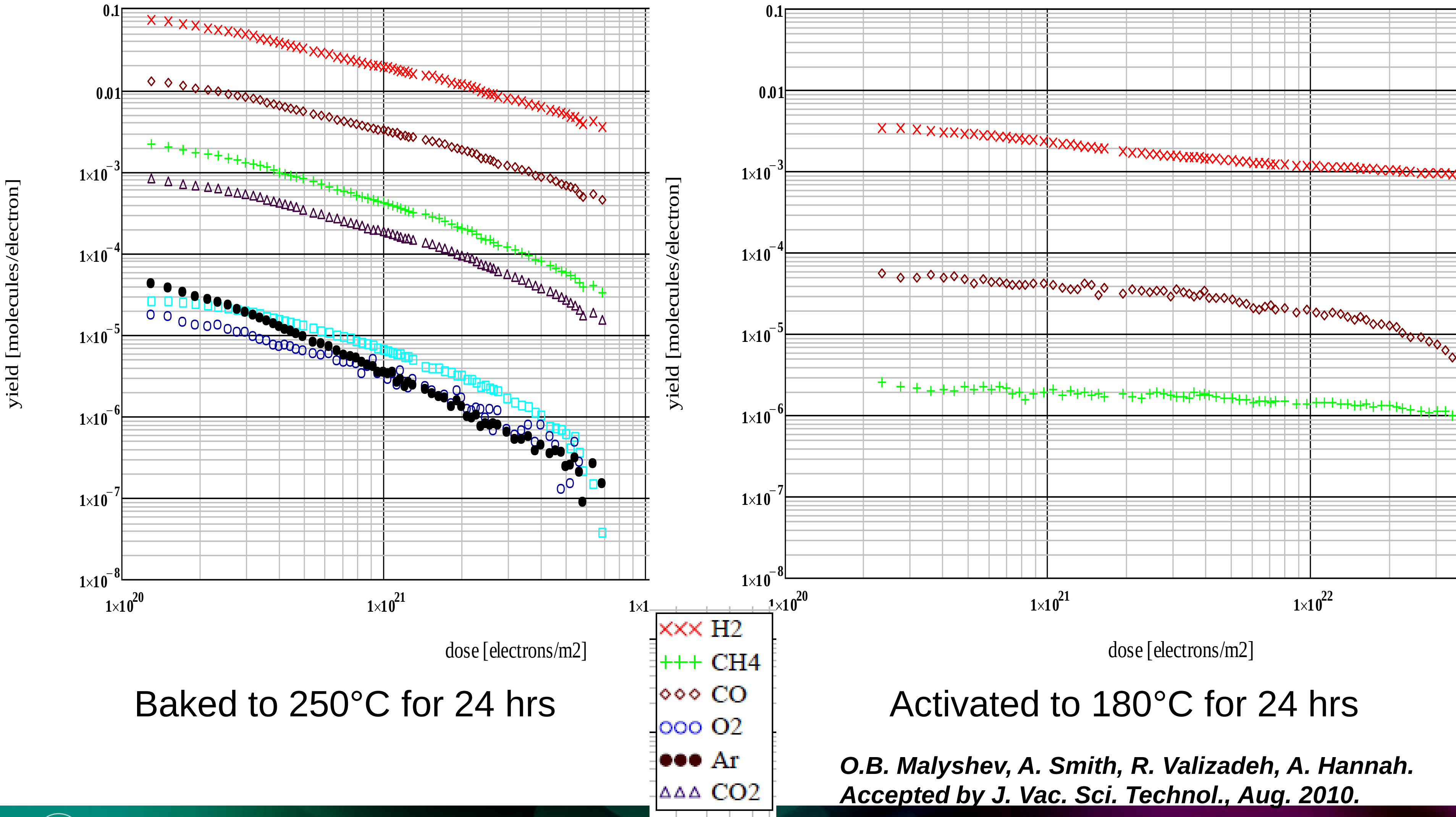
ESD: stainless steel vs non-activated NEG coated vacuum chamber



Baked to 250°C for 24 hrs
Pumped for 30 days

Baked to 80°C for 24 hrs
pumped for 1 day

ESD: stainless steel vs activated NEG coated vacuum chamber



Baked to 250°C for 24 hrs

Activated to 180°C for 24 hrs

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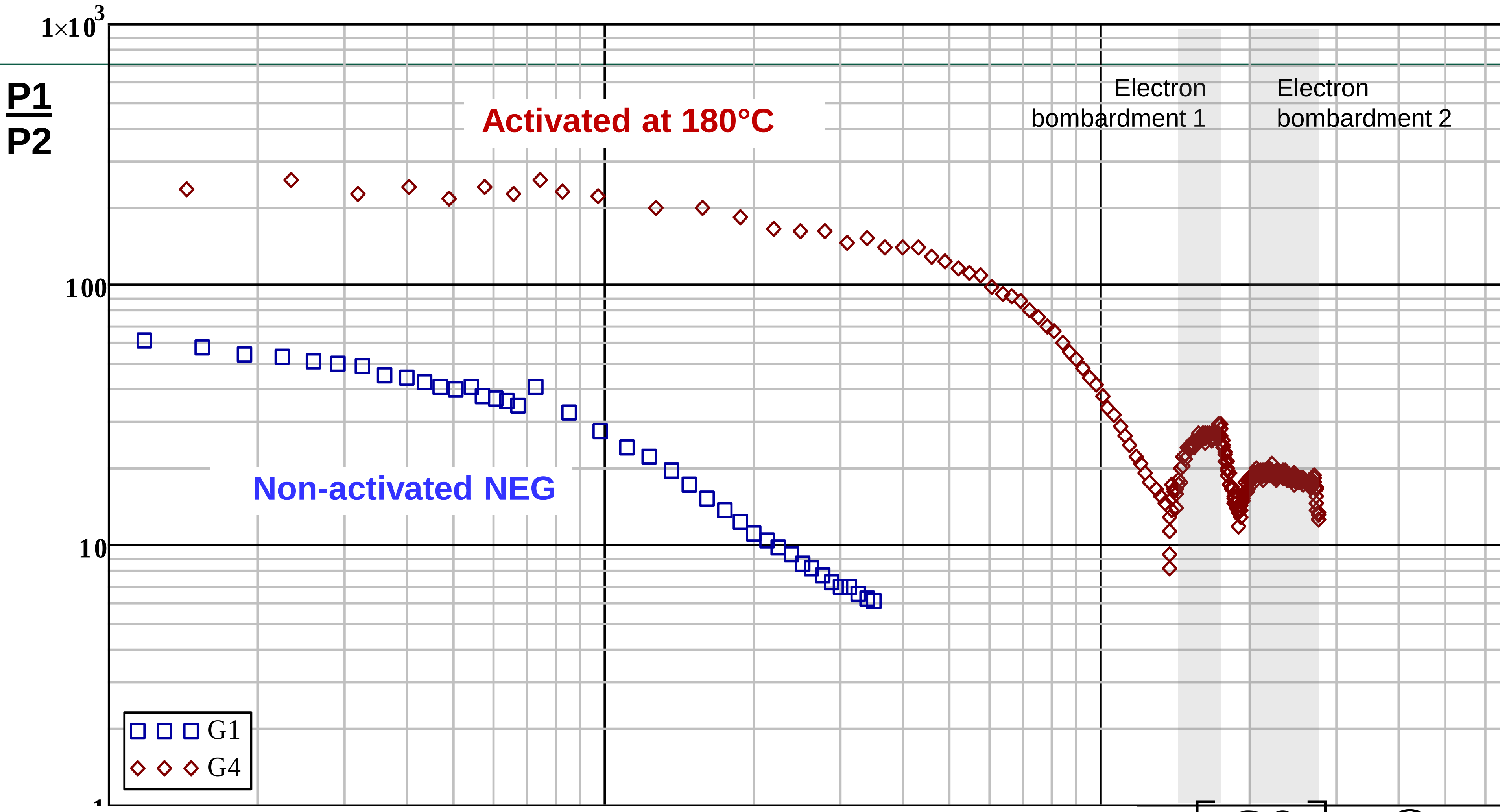
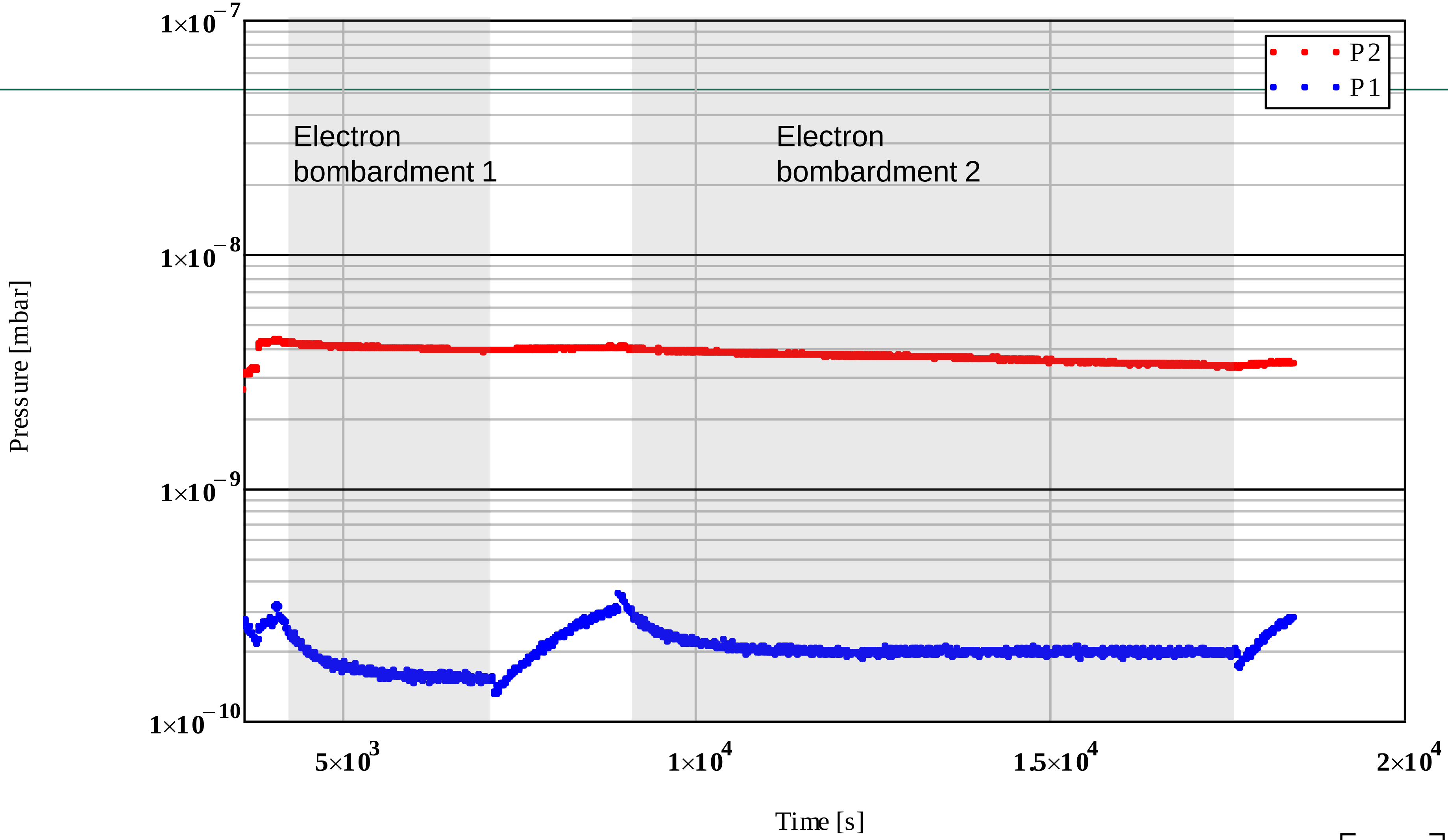
Electron stimulated NEG activation



The electron stimulated NEG activation efficiency estimated as **$7.9\times10^{-4} < \sigma_1 < 2.4\times10^{-3}$ [CO/e^-]**

$$\sigma_1 \left[\frac{CO}{e^-}\right] = \frac{\Omega_{CO}}{D}$$

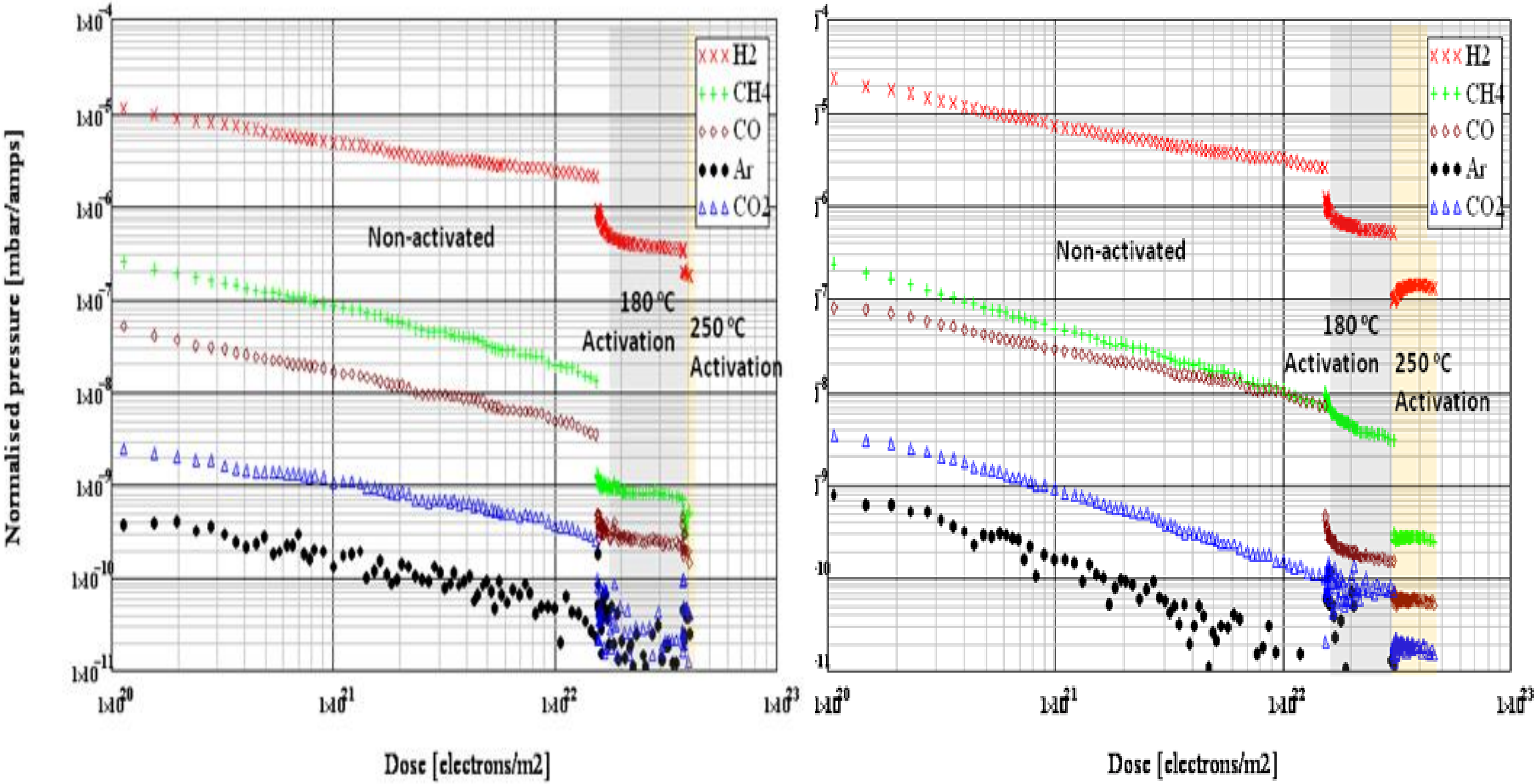
Electron stimulated NEG activation



The electron stimulated NEG activation efficiency estimated as

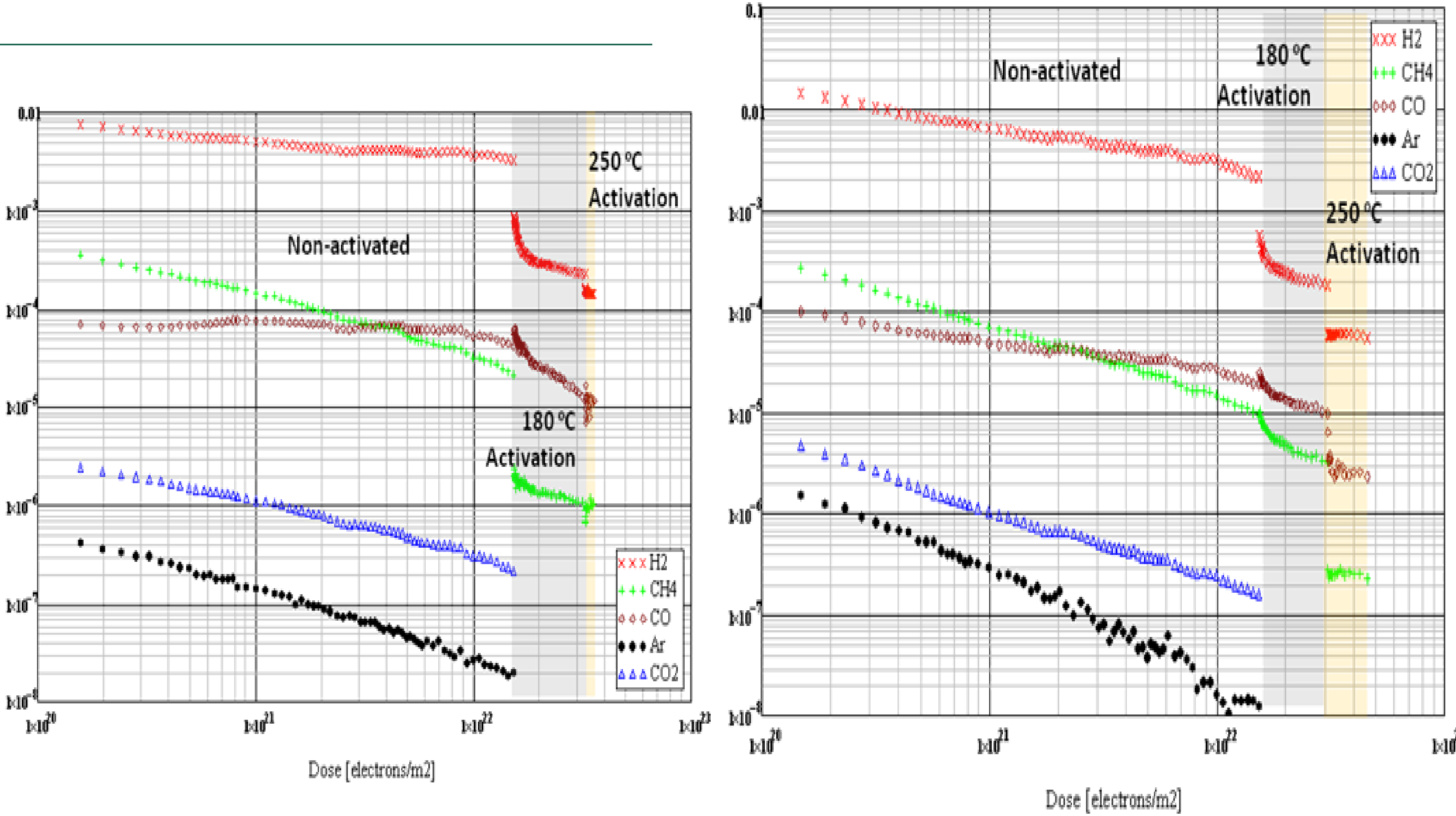
$$\sigma_2 = \frac{Q_{CO}}{k_B T}\frac{q_e}{I} = 2.2\times10^{-3}\left[\frac{CO}{e^-}\right]$$

Normalised pressure P1
Columnar vs. Dense

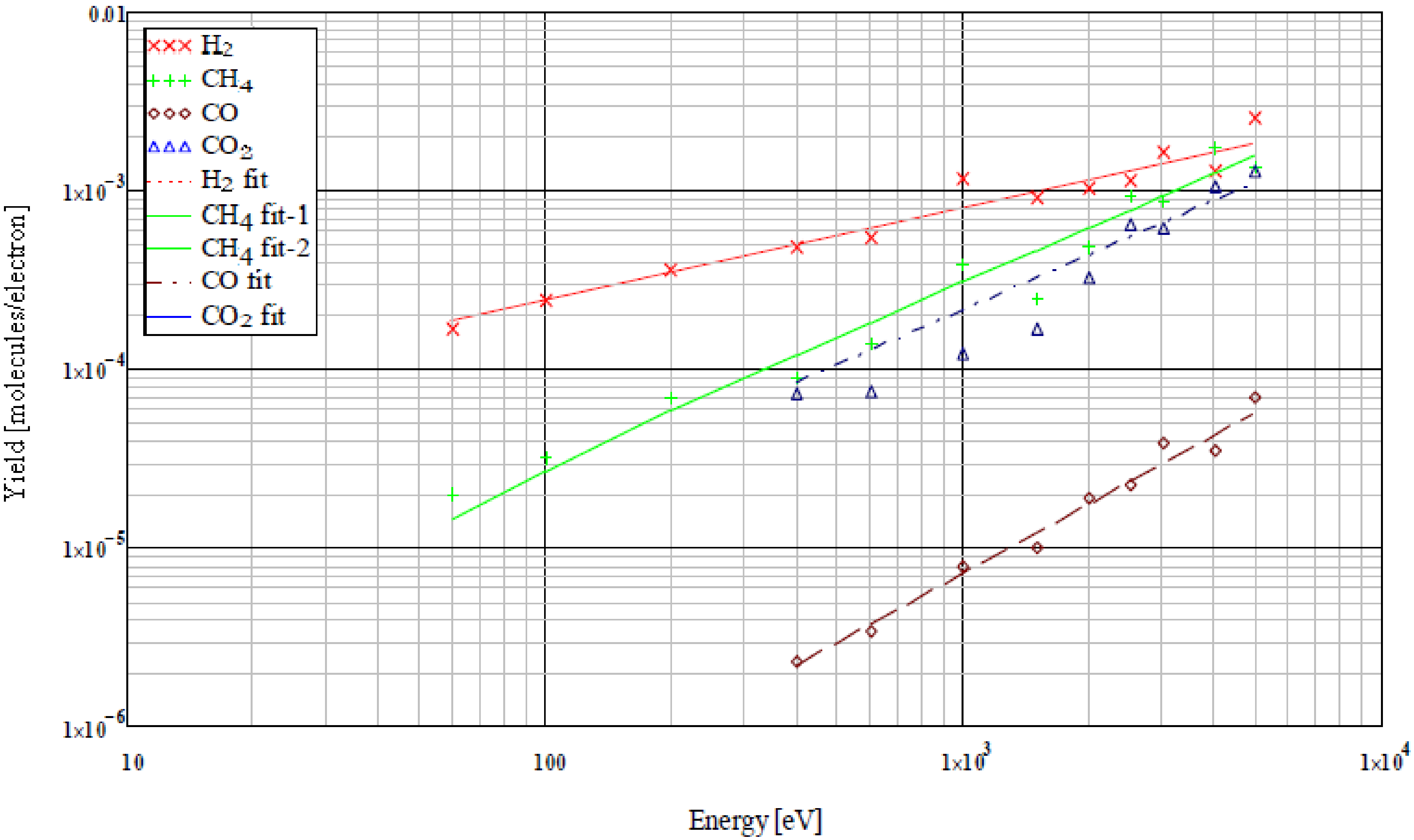


$$\alpha_c(H_2) = 1.5\ \alpha_d(H_2);\ \alpha_c(CO) = 1.5\ \alpha_d(CO)$$

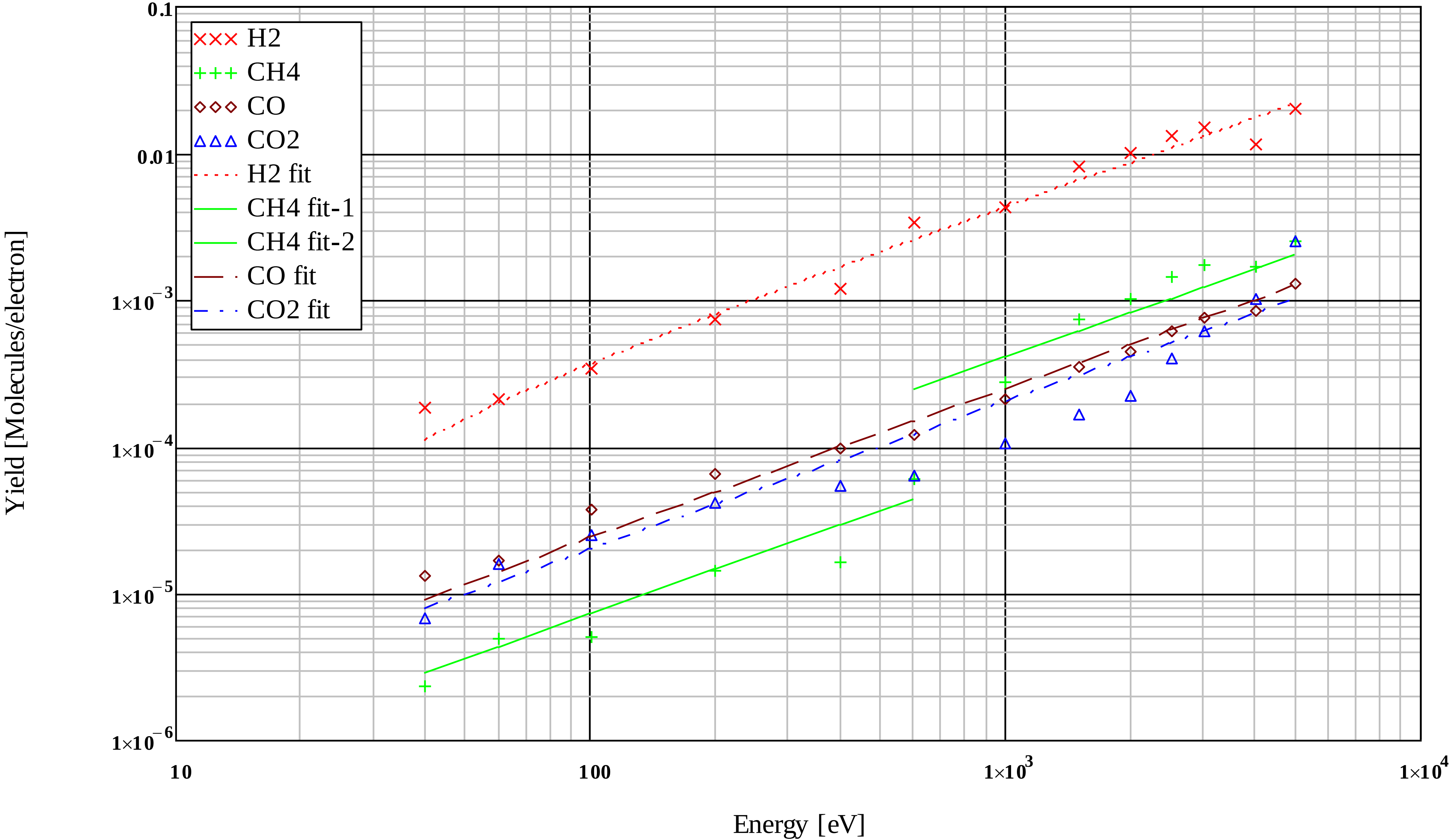
ESD Yields
Columnar vs. Dense



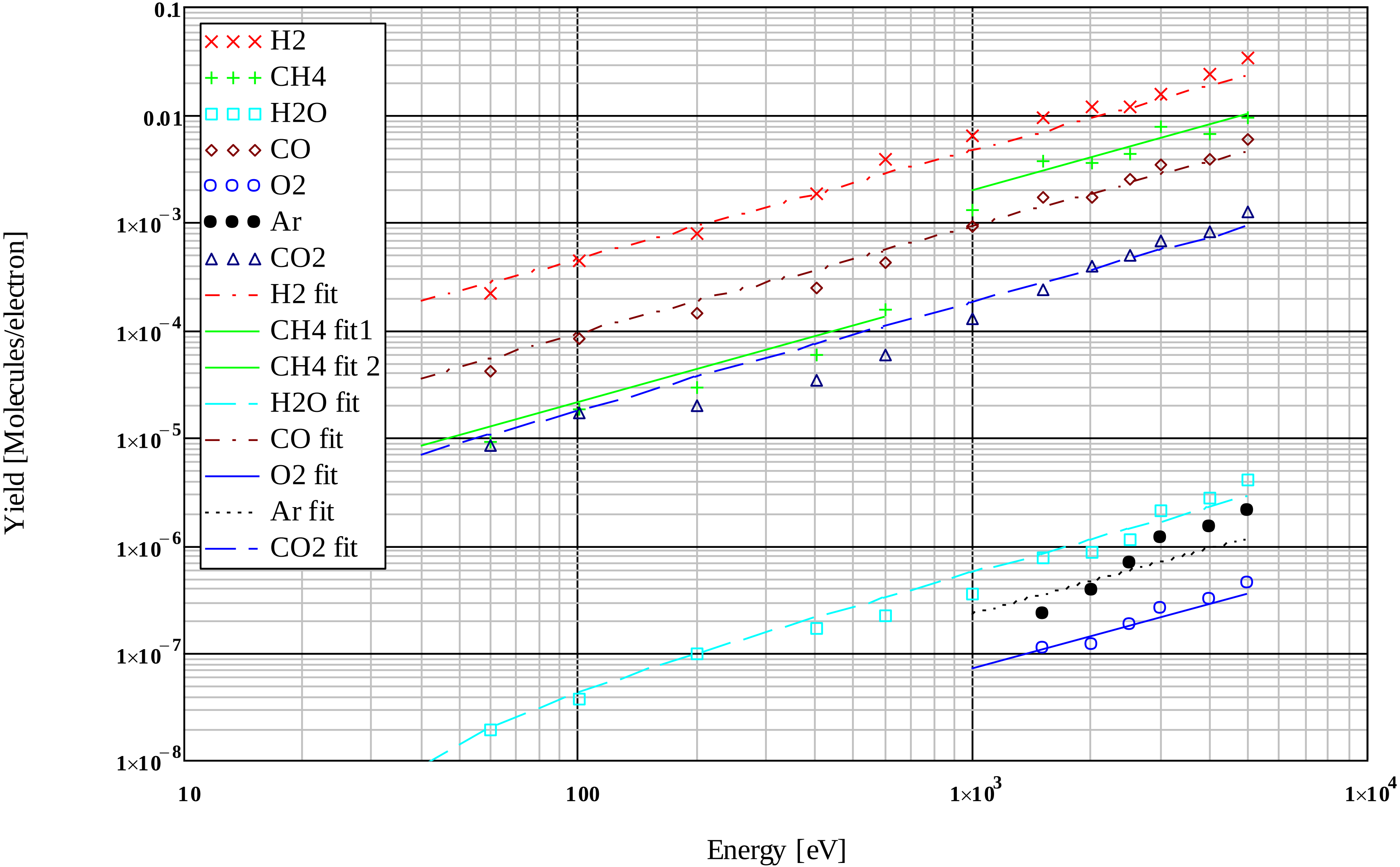
$\eta(E_{e-})$ for different gases for NEG coating



$\eta(E_{e-})$ for different gases for 316LN



$\eta(E_{e-})$ for different gases for aluminium alloy



Conclusions:

- ASTeC activation procedure minimises NEG poisoning from non-coated vacuum chamber components
- Role of element:
  - Zr-based – highest sticking probability and capacity, lowers activation temp.
  - Ti-based – lowest sticking probability and capacity, highest activation temp
- Role of grain size
  - Activation temperature reduces with a grain size die to increase the grain boundary density
- Quaternary alloy demonstrated the lowest activation temperature and best pumping properties;
  - Pure Zr film is good as well
- Alloy target is better than twisted wires
- The improvement and further development of NEG coatings requires
  - Intensive use surface analysis techniques
  - Evaluation under photon, electron and ion bombardment.

Conclusions (2):

- An ESD set-up for tubular sample
  - Uniform bombardment along the tube
  - From both pumping and non-pumping samples.
- The ESD yields as a function of electron dose :
  - 316L stainless steel sample after bakeout at 250°C
  - Ti-Zr-V coated before NEG activation and after activation at 180°C and 250 °C.
  - Desorption yields from SS are comparable with earlier results from literature;
  - The initial desorption yields from NEG coating are 20 times lower for H_2, 1000 times lower for CH_4 and 200 times lower for CO, the desorption yields for other gases below the installation sensitivity.
- The ESD yields as a function of electron energy:
  - were measured in the energy range between 40 eV and 5 keV.
  - a linear dependence was measured for most of gases
  - except for H_2, for which the dependence is: $\eta(E) \propto E^{2/3}$
- The electron bombardment induced pumping of the CO saturated NEG film was observed for a first time
  - this effect is similar to photon induced pumping of the NEG film observed earlier.

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Co-authors (team):

ASTeC .

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